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Field Measurements of Full-Scale Hazardous Waste Treatment Facilities - Organic Solvent Wastes

Metcalf and Eddy, Inc., Wakefield, MA

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16. ABSTRACT

This report describes the treatment of organic hazardous wastes by distillation, thin-film evaporation, incineration, steam stripping, waste blending, carbon adsorption and activated sludge at full-scale facilities.

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FIELD MEASUREMENTS OF FULL-SCALE HAZARDOUS WASTE TREATMENT FACILITIES - ORGANIC SOLVENT WASTES

By

Metcalf & Eddy Engineers, Inc.
Wakefield, MA 01880

Contract No. 68-03-3166

Project Monitor

Ronald J. Turner
Water and Hazardous Waste Treatment Research Division
Risk Reduction Engineering Laboratory
Cincinnati, Ohio 45268

RISK REDUCTION ENGINEERING LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

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FOREWORD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of materials that, if improperly dealt with, can threaten both public health and the environment. The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. These laws direct the EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Risk Reduction Engineering Laboratory is responsible for planning, implementing, and managing research, development, and demonstration programs to provide an authoritative, defensible engineering basis in support of the policies, programs, and regulations of the EPA with respect to drinking water, wastewater, pesticides, toxic substances, solid and hazardous wastes, and Superfund-related activities. This publication is one of the products of that research and provides a vital communication link between the researcher and the user community.

This report provides the results of field evaluations at full-scale hazardous waste treatment facilities which process organic and solvent waste streams. Those wishing additional information on these evaluations are urged to contact the EPA Project Officer.

E. Timothy Oppelt, Acting Director
Risk Reduction Engineering Laboratory

ABSTRACT

Treatment of organic wastes by full scale treatment facilities was evaluated through field evaluations of offsite hazardous waste treatment facilities to provide data regarding alternative treatment technologies to land disposal and the applicability of these technologies to specific waste streams. Field sampling and analysis provided characterization of the waste, product and residues associated with technologies such as distillation, steam distillation, thin film evaporation, heated screw auger evaporation, incineration, steam stripping, waste blending, carbon adsorption, and activated sludge treatment at the facilities tested. An evaluation of treatment effectiveness based on waste, product, and residue characteristics and a discussion on residue management were prepared for each facility.

This report was submitted in fulfillment of Contract No. 68-03-3166 by Metcalf and Eddy Engineers, Inc., under the sponsorship of the U.S. Environmental Protection Agency. This report covers tests conducted from June 1985 to September 1986.

CONTENTS

Page

ABSTRACT iv

LIST OF TABLES x

LIST OF FIGURES xii

SECTION 1. INTRODUCTION 1-1
  PROJECT BACKGROUND 1-1
  OBJECTIVES 1-2
  SCOPE 1-2

SECTION 2. CONCLUSIONS 2-1
  BATCH DISTILLATION AND STEAM DISTILLATION 2-1
  AGITATED THIN FILM EVAPORATION 2-2
  HEATED SCREW AUGER 2-2
  INCINERATION 2-3
  WASTE BLENDING/FUEL SUBSTITUTION 2-3
  STEAM STRIPPING 2-3
  CARBON ADSORPTION 2-4
  ACTIVATED SLUDGE 2-4

SECTION 3. PROGRAM OVERVIEW 3-1
  REGULATORY REVIEW 3-1
  SELECTION OF ORGANIC WASTES AND TREATMENT PROCESSES FOR EVALUATION 3-3
  SITE SELECTION PROCESS 3-7
  SAMPLING PROGRAM 3-8

SECTION 4. SELECTED ALTERNATIVE TREATMENT TECHNOLOGIES FOR ORGANIC HAZARDOUS WASTES 4-1

SECTION 5. BATCH DISTILLATION 5-1
  THEORY 5-1
  FACILITY A 5-3
    Process Description 5-3
    Sampling Program 5-7
    Data 5-7
      Waste Characterization 5-7
      Product Characterization 5-15
      Residue Characterization 5-15
    Evaluation of Effectiveness 5-16
    Residue Management 5-18

Page

FACILITY D.. 5-18
Process Description... 5-18
Sampling Program.. 5-21
Data.. 5-22
Waste Characterization.. 5-22
Product Characterization.. 5-24
Residue Characterization.. 5-27
Evaluation of Effectiveness....................................... 5-29
Overall Treatment System.. 5-29
Main Still.. 5-29
Solvent Process Tank.. 5-30
Batch Kettle.. 5-30
Batch Still Reboiler.. 5-30
Thinning Tank... 5-31
Residue Management.. 5-31

SECTION 6. STEAM DISTILLATION..................................... 6-1
THEORY.. 6-1
FACILITY C.. 6-2
Process Description... 6-2
Sampling Program.. 6-7
Data.. 6-12
Waste Characterization.. 6-12
Product Characterization.. 6-12
Residue Characterization.. 6-20
Evaluation of Effectiveness....................................... 6-21
Residue Management.. 6-22

SECTION 7. AGITATED THIN FILM EVAPORATION AND SCREW AUGER EVAPORATION.... 7-1
THEORY.. 7-1
Agitated Thin Film Evaporator..................................... 7-1
Heated Screw Auger.. 7-3
FACILITY J.. 7-3
Process Description... 7-3
Sampling Program.. 7-6
Data.. 7-6
Waste Characterization.. 7-8
Product Characterization.. 7-8
Residue Characterization.. 7-8
Evaluation of Effectiveness....................................... 7-9
Residue Management.. 7-10

FACILITY K.. 7-10
Process Description - ATFE.. 7-10
Sampling Program.. 7-12
Data.. 7-12
Waste Characterization.. 7-12
Product Characterization.. 7-15
Residue Characterization.. 7-15

Page

Evaluation of Effectiveness.................................. 7-16
Residue Management... 7-17
Process Description - Heated Screw Auger Evaporation......... 7-17
Sampling Program... 7-19
Data... 7-19
  Waste Characterization..................................... 7-19
  Product Characterization................................... 7-19
  Residue Characterization................................... 7-22
Evaluation of Effectiveness.................................. 7-22
Residue Management... 7-23

FACILITY L.. 7-23
  Process Description... 7-23
  Sampling Program.. 7-24
  Data.. 7-24
    Waste Characterization...................................... 7-24
    Product Characterization.................................... 7-24
    Residue Characterization.................................... 7-28
  Evaluation of Effectiveness................................... 7-28
  Residue Management.. 7-28

SECTION 8. INCINERATION.. 8-1
  THEORY... 8-1
  PROCESS DESCRIPTIONS... 8-2
    Facility F... 8-2
    Feed... 8-4
    Residuals.. 8-4
    Facility H... 8-6
  SAMPLING PROGRAMS.. 8-8
    Facility F... 8-8
    Facility H... 8-8
  DATA... 8-14
    Waste Characterization... 8-14
      Facility F... 8-14
      Facility H... 8-14
    Residue Characterization... 8-19
      Facility F... 8-19
      Facility H... 8-25
  EVALUATION OF EFFECTIVENESS.. 8-25
  RESIDUE MANAGEMENT... 8-28

SECTION 9. WASTE BLENDING/FUEL SUBSTITUTION.......................... 9-1
  THEORY... 9-1
  PROCESS DESCRIPTION.. 9-2
  SAMPLING PROGRAM... 9-2
  DATA... 9-2
    Waste Characterization... 9-2
    Product Characterization... 9-7
    Residue Characterization... 9-7

Page

EVALUATION OF EFFECTIVENESS........ 9-11
RESIDUE MANAGEMENT........ 9-12

SECTION 10. STEAM STRIPPING........ 10-1
- THEORY........ 10-1
- FACILITY G........ 10-5
  - Process Description........ 10-5
  - Sampling Program........ 10-5
  - Data........ 10-7
    - Waste Characterization........ 10-7
    - Product Characterization........ 10-7
    - Residue Characterization........ 10-7
  - Evaluation of Effectiveness........ 10-11
  - Residue Management........ 10-11
- FACILITY M........ 10-11
  - Process Description........ 10-11
  - Sampling Program........ 10-11
  - Data........ 10-13
    - Waste Characterization........ 10-13
    - Product Characterization........ 10-13
    - Residue Characterization........ 10-13
  - Evaluation of Effectiveness........ 10-13
  - Residue Management........ 10-15
- FACILITY N........ 10-15
  - Process Description........ 10-15
  - Sampling Program........ 10-15
  - Data........ 10-17
    - Waste Characterization........ 10-17
    - Product Characterization........ 10-17
    - Residue Characterization........ 10-17
  - Evaluation of Effectiveness........ 10-19
  - Residue Management........ 10-19

SECTION 11. ACTIVATED SLUDGE TREATMENT........ 11-1
- THEORY........ 11-1
- PROCESS DESCRIPTION........ 11-1
- SAMPLING PROGRAM........ 11-5
- DATA........ 11-10
  - Waste Characterization........ 11-10
    - Methanol Still Bottoms........ 11-10
    - Influent to Aeration........ 11-10
    - AFI Waste........ 11-21
    - Secondary Effluent........ 11-21
    - Waste Activated Sludge........ 11-22
- EVALUATION OF EFFECTIVENESS........ 11-22
- RESIDUE MANAGEMENT........ 11-22

Page

SECTION 12. CARBON ADSORPTION.................................... 12-1
THEORY.. 12-1
FACILITY B.. 12-2
Process Description....................................... 12-2
Sampling Program.. 12-7
Data.. 12-8
Influent Characterization................................. 12-8
Effluent Characterization................................. 12-8
Evaluation of Effectiveness............................... 12-11
FACILITY C.. 12-11
Process Description....................................... 12-11
Sampling Program.. 12-13
Data.. 12-13
Influent Characterization................................. 12-13
Effluent Characterization................................. 12-13
Evaluation of Effectiveness............................... 12-13

LIST OF TABLES

Number Page

1 HAZARDOUS WASTES REGULATED BY LAND DISPOSAL RESTRICTIONS OF SOLVENTS 3-4
2 ANALYTICAL AND SAMPLE PREPARATION METHODS AND REFERENCES 3-9
3 Treatment Technology/Waste Stream/Facility Matrix 4-2
4 FACILITY A SAMPLE POINTS AND SAMPLING METHODS 5-8
5 GENERATOR'S MANIFEST DATA FOR WASTE SOLVENTS 5-9
6 BATCH DISTILLATION ANALYTICAL DATA - BATCH 1 FACILITY A 5-10
7 BATCH DISTILLATION ANALYTICAL DATA - BATCH 2 FACILITY A 5-11
8 BATCH DISTILLATION ANALYTICAL DATA - BATCH 3 FACILITY A 5-12
9 BATCH DISTILLATION ANALYTICAL DATA - BATCH 4 FACILITY A 5-13
10 SAMPLE LOCATIONS AND PROCEDURES - FACILITY D 5-23
11 MAIN STILL ANALYTICAL RESULTS - FACILITY D 5-25
12 BATCH DISTILLATION RESULTS - FACILITY D 5-26
13 BATCH STILL REBOILER RESULTS - FACILITY D 5-28
14 FACILITY C SAMPLE LOCATIONS AND PROCEDURES 6-8
15 ORGANIC SOLVENTS TREATED DURING SAMPLING PROGRAM 6-13
16 ANALYTICAL DATA - STEAM DISTILLATION - NON CHLORINATED DAY 1 6-14
17 ANALYTICAL DATA - STEAM DISTILLATION - NON CHLORINATED DAY 2 6-16
18 ANALYTICAL DATA - STEAM DISTILLATION CHLORINATED DAY 3 6-18
19 ATFE - WASTE CHARACTERIZATION 7-2
20 FACILITY J ANALYTICAL RESULTS 7-7
21 FACILITY K - THIN FILM EVAPORATOR ANALYTICAL RESULTS 7-14
22 FACILITY K - HEATED SCREW AUGER ANALYTICAL DATA SUMMARY 7-20
23 FACILITY K - SCREW AUGER EP TOXICITY RESULTS BY CURRENT AND PROPOSED PROCEDURES 7-21
24 FACILITY L - THIN FILM EVAPORATOR ANALYTICAL RESULTS 7-26
25 FACILITY L - TCLP DATA FOR THE STILL BOTTOMS DISCHARGED FROM THE TFE 7-29
26 FACILITY F - SAMPLE LOCATIONS AND PROCEDURES 8-9
27 FACILITY H - SAMPLE LOCATIONS AND PROCEDURES 8-12
28 FACILITY F - WASTE CHARACTERIZATION 8-15
29 FACILITY H - WASTE CHARACTERIZATION 8-17
30 FACILITY F - ASH ANALYTICAL DATA - ORGANICS 8-20
31 FACILITY F - ASH ANALYTICAL DATA - METALS 8-21
32 FACILITY F - SCRUBBER WASTEWATER TREATMENT DATA 8-22
33 FACILITY F - EFFLUENT METALS 8-24
34 FACILITY H - ASH ANALYTICAL DATA - METALS 8-26
35 FACILITY H - SCRUBBER WASTEWATER DATA 8-27
36 APPROXIMATE MASS BALANCE - FACILITY F 8-29
37 FACILITY B - SAMPLING LOCATIONS AND PROCEDURES 9-4
38 ORGANIC WASTE INCORPORATED INTO FUEL BLENDS 9-5
39 FACILITY B - FUEL BLENDING PROCESS DATA 9-6
40 FACILITY B - NON PUMPABLE SLUDGE TCLP DATA 9-9
41 PROCESS PARAMETERS AND WASTE STREAM PROFILES FOR FACILITIES G, M, AND N STEAM STRIPPERS 10-2
42 FACILITY G - SAMPLE LOCATION DESCRIPTIONS 10-8
43 FACILITY G - PHYSICAL AND CHEMICAL SAMPLE FRACTIONS 10-9

Number Page

44 FACILITY G - ANALYTICAL DATA.................................. 10-10
45 FACILITY M - ANALYTICAL DATA.................................. 10-14
46 FACILITY N - ANALYTICAL DATA.................................. 10-18
47 FACILITY I - SAMPLING LOCATIONS AND PROCEDURES.................. 11-6
48 FACILITY I - SUMMARY OF ANALYTICAL DATA OF DISCRETE SAMPLES... 11-11
49 FACILITY I - INFLUENT TO AERATION GRAB SAMPLES - DAY 1........... 11-12
50 FACILITY I - INFLUENT TO AERATION GRAB SAMPLES - DAY 2........... 11-13
51 FACILITY I - INFLUENT TO AERATION GRAB SAMPLES - DAY 3........... 11-14
52 FACILITY I - AFI GRAB SAMPLES - DAY 1........................... 11-15
53 FACILITY I - AFI GRAB SAMPLES - DAY 2........................... 11-16
54 FACILITY I - AFI GRAB SAMPLES - DAY 3........................... 11-17
55 FACILITY I - SECONDARY EFFLUENT GRAB SAMPLES - DAY 1............ 11-18
56 FACILITY I - SECONDARY EFFLUENT GRAB SAMPLES - DAY 2............ 11-19
57 FACILITY I - SECONDARY EFFLUENT GRAB SAMPLES - DAY 3............ 11-20
58 FACILITY B - SAMPLING LOCATIONS AND PROCEDURES.................. 12-8
59 FACILITY B - CARBON ADSORPTION DATA............................. 12-9
60 FACILITY C - SAMPLING LOCATIONS AND PROCEDURES.................. 12-14
61 FACILITY C - CARBON ADSORPTION DATA............................. 12-15

LIST OF FIGURES

Number Page

1 Batch Distillation Schematic.... 5-2
2 Flow Diagram/Sample Points for Batch Distillation at Facility A.... 5-5
3 Facility D Distillation Process.... 5-19
4 Hausbrand Diagram for 1,1,1-trichloroethane at 1 ATM (760 mm Hg).... 6-3
5 Flow Diagram/Sample Points for Steam Distillation at Facility C, Nonchlorinated Solvents.... 6-4
6 Flow Diagram/Sample Points for Steam Distillation at Facility C, Chlorinated Solvents.... 6-5
7 Thin Film Evaporator at Facility J.... 7-4
8 Thin Film Evaporator Process Schematic - Facility K.... 7-13
9 Screw Auger Process Schematic - Facility K.... 7-18
10 Thin Film Evaporation Process Schematic - Facility L.... 7-25
11 Facility F Process Flow Diagram.... 8-3
12 Facility F Scrubber Wastewater Treatment.... 8-5
13 Facility H Process Flow Diagram.... 8-7
14 Flow Diagram/Sample Points for Organic Waste Blending at Facility B.... 9-3
15 Settleability Analysis for the Organic Waste Blending Program at Facility B.... 9-8
16 Steam Stripper Schematic.... 10-3
17 Facility G - Methylene Chloride Steam Stripping Schematic.... 10-6
18 Facility M Steam Stripping Schematic and Sampling Points.... 10-12
19 Facility N Steam Stripping Schematic and Sampling Points.... 10-16
20 Activated Sludge Process Schematic.... 11-2
21 Biological Wastewater Treatment Plant - Facility I.... 11-4
22 Schematic of Fixed Bed Adsorption System.... 12-3
23 Typical Carbon Breakthrough Curve.... 12-4
24 Facility B Flow Diagram/Sample Points for Aqueous Waste Treatment System.... 12-6
25 Flow Diagram/Sample Points for Aqueous Waste Treatment at Facility C.... 12-12

SECTION 1

INTRODUCTION

This report discusses the treatment of organic hazardous wastes at full scale facilities. Treatment performance has been measured by field sampling and on-site process monitoring data. Metcalf and Eddy, Inc. conducted sampling programs at eight treatment, storage and disposal facilities (TSDF's) managing hazardous wastes. Evaluations of three additional TSDF's sampled by another RREL contractor, Alliance Technologies Corp., (formerly GCA Technology Division) and two additional sites sampled by Research Triangle Institute are also included. Only those data relevant to organic solvent treatment are presented in this report.

PROJECT BACKGROUND

Since 1980, the Environmental Protection Agency (EPA) has issued regulations under the Resource Conservation and Recovery Act (RCRA) restricting the land disposal of hazardous waste. On November 8, 1984, Congress amended RCRA through the Hazardous and Solid Waste Amendments (HSWA) of 1984 to further limit the land disposal of hazardous waste. The amendments require that EPA ban the land disposal of certain hazardous wastes unless it is determined that the ban is not necessary to protect human health and the environment. EPA is required to determine treatment methods or levels and existing treatment capacity according to schedules set by the amendments. The schedules prioritize hazardous waste categories to be addressed. If schedules are not met, the wastes will be banned automatically from further land disposal.

EPA's Office of Solid Waste (OSW) has been given the primary responsibility for developing the regulations required by HSWA. OSW has involved EPA's Office of Research and Development (ORD) in the program "Assessment of Alternative Technologies" to treat hazardous wastes. This program is investigating the extent to which land disposal is currently used for hazardous wastes that are being considered for the land disposal ban. The program is also identifying alternatives to land disposal. Alternatives include treatment, recycling, and source reduction. The program is focusing on the performance, available and future capacity, and environmental effects of proven technologies.

Metcalf & Eddy, Inc., and Alliance Technologies Corporation conducted parallel test programs to evaluate full-scale treatment processes at commercial off-site hazardous waste treatment, storage and disposal facilities (TSDF's). This Summary Report is a compilation of the results from each Facility Test Report related to organic waste streams.

OBJECTIVES

The objective in conducting this project is to generate data documenting waste treatment practices at offsite treatment, storage, and disposal facilities. The following specific objectives were addressed:

- identify priority waste streams needing additional evaluations;
- identify alternative technologies to treat priority waste streams;
- develop a specific approach to measure performance of alternative technologies;
- conduct sampling programs at treatment facilities;
- document field activities and present data in summary reports for each facility;
- summarize data generated regarding alternative treatment technologies.

SCOPE

Several EPA-Office of Solid Waste reports on the technical assessment of available treatment and disposal alternatives for specific wastes containing corrosives, metals and/or cyanides, waste solvents and halogenated organics were compiled in support of this program.

1. "Technical Assessment of Treatment Alternatives for Wastes Containing Corrosives" by Camp Dresser and McKee, Inc.
2. "Technical Assessment of Treatment Alternatives for Wastes Containing Metals and/or Cyanides" by Versar.
3. "Supplemental Report on the Technical Assessment of Treatment Alternatives for Waste Solvents" by Engineering-Science.
4. "Assessment of Treatment Practices for Proposed Hazardous Waste-Listings F020, F021, F022, F023, F026, F027, and F028" by Radian Corporation.
5. "Technical Assessment of Treatment Alternatives for Wastes Containing Halogenated Organics" by GCA Corporation.

These documents were reviewed to evaluate existing data and to identify areas requiring further data development. The technologies selected to be tested were based upon the areas identified where additional information was needed. Field testing was conducted to expand the existing database. The facilities were selected through a site selection process which involved site visits to candidate facilities and a determination of which facilities best fit the waste type and technology criteria set forth in the objectives of this study. The discussion of the wastes and technologies to be considered and a description of the site selection process are contained in Section 3, "Program Overview." Section 4 of this report includes a summary of the waste types and treatment processes that have been evaluated in this report. Subsequent sections (Sections 5-12) discuss the treatment technologies and the results from the field evaluations of these technologies at individual facilities. These data will provide EPA with information on the performance of specific unit operations on specific waste streams. Information was also collected to evaluate environmental impacts (e.g. air emissions, sludge disposal, effluent discharge) from these technologies. Facility test reports were prepared for each sampling episode.

The data obtained during this study also will be of interest to generators of organic hazardous waste streams.

This report is a summary of all data collected on the treatment of organic waste streams.

Because standards restricting the land disposal of hazardous wastes have been promulgated since this study, current waste disposal practices at these facilities may not be the same as described herein.

SECTION 2

CONCLUSIONS

The alternative treatment technologies evaluated during this study were effective in treating organic waste streams to recover organic solvents, produce residues to be used as fuel blends and reduce levels of hazardous constituents. The effectiveness of the following treatment technologies and the conclusions drawn are based on a limited number of facility tests. Because standards restricting the land disposal of hazardous waste have been promulgated since this study, current waste disposal practices at these facilities may not be the same as described herein.

BATCH DISTILLATION AND STEAM DISTILLATION

1. Simple batch distillation is applicable to treatment and recovery of hazardous solvent wastes where the impurities that render these solvents unsuitable for further direct use are high boiling materials or nonvolatile solids.

2. Facility A treated both halogenated and non-halogenated waste streams. The wastes included methylene chloride, isopropanol, 1,1,1-trichloroethane, and methyl ethyl ketone. Facility D treated wastes containing methyl ethyl ketone mixed with paint pigments, oil water emulsion and mixed solvent wastes.

3. The still bottoms produced at Facility A generally had a high BTU, low chlorine content and were suitable for fuel blending. Only one batch, from treatment of 1,1,1-trichloroethane waste, was disqualified for use as a fuel due to high chlorine content. The methylene chloride batch was retained to be redistilled even though the chlorine content was low and there was sufficient heating value for a fuel blend, because a large amount of solvent remained in the bottoms product. Both bottoms products which were acceptable for fuel blending were the residues from the two non-halogenated waste batches. At Facility A residues which can't be used in fuel blends are solidified prior to being landfilled. Of the residues available to be sampled at Facility D, the main still produced a residue with a high BTU, low water and low chlorine content suitable for use as a fuel except that the presence of heavy metals precludes its uses as a fuel thus the bottoms had to be drummed for disposal. The batch still reboiler residue had a high water content. At Facility D all still bottom residues produced were reportedly heated until solid and then drummed for disposal. From the results of Facility A and D distillation appears to be able to produce a high BTU, low chlorine content residue suitable for fuel blending in some cases. In some cases bottoms had a high chlorine content or contained heavy metals which rendered the residues unsuitable for fuel blending.

4. Solvent product recovered at both facilities were generally crude product cuts which could be sold for use in applications that do not require pure solvents. At Facility A, 52 percent of the 1543 gallons of solvent laden wastes was recovered as solvent product.

5. Distillation was effective in reducing the quantity of material for landfill disposal by recovering the waste solvent.

6. At Facility D, along with recovering solvent, the distillation process generated a wastewater stream containing some organic contaminants which presents an additional stream which must be treated.

7. The steam distillation unit at Facility C treated both halogenated and non-halogenated organic solvents and recovered these organics in the distillate but produced a solvent laden wastewater which is a notable disadvantage of direct steam distillation and steam stripping.

AGITATED THIN FILM EVAPORATION

8. The thin film evaporator is effective at recovering non-halogenated solvents and in producing still bottoms that can be utilized as supplemental fuel.

9. Halogenated solvents can also effectively be recovered by thin film evaporation but the bottoms use as a fuel supplement is dependent on the residual chlorine content. High chlorine content bottoms are not desirable for fuel blending due to the possibility of formation of HCl during combustion.

10. The thin film evaporator concentrates metals in the still bottoms.

11. In order to keep the still bottoms pumpable some solvent needs to remain in the bottoms and therefore the residual solvent concentrations could remain high and limit solvent recovery.

HEATED SCREW AUGER

12. At Facility K, the heated screw auger achieved significant reductions in waste solvent concentration for non-pumpable solid waste and pumpable organic sludges.

13. Residual stream generated had a low heat value thus it is not used for fuel blending, etc.

14. Metals were concentrated in the residual stream.

INCINERATION

15. Facility F incinerated organic wastes containing common solvent compounds such as acetone, benzene, chloroform, methylene chloride, methyl ethyl ketone, tetrachloroethylene, toluene, trichloroethylene and xylene. The ash did contain several organic compounds which may be indicative of incomplete combustion due to inadequate retention time, mixing or temperature in the incinerator. A rough mass balance indicates greater than 99.93 percent destruction. Facility H's waste contained a large concentration of the volatile organic compound toluene along with several semivolatile organic compounds all of which were not detected in the ash.

16. Properly designed and operated incineration systems can achieve virtually complete destruction of organic compounds.

17. At Facility F and H, metals tend to concentrate in the treatment residues - ash and scrubber wastewater since metals are not destroyed by incineration.

18. The scrubber wastewater for both facilities contained no detectable quantities of organic compounds.

WASTE BLENDING/FUEL SUBSTITUTION

19. Waste blending is effective at producing fuel blends from organic wastes. This includes solid organic wastes which are used as substitutes for conventional fuels in high temperature industrial processes such as industrial boilers and kilns.

20. Wastes which are unacceptable for fuel substitution due to low heating value, high chlorine concentration, high ash content, or high viscosity may be blended with higher quality organic wastes or with fuel oil to produce a mixture of acceptable quality for fuel substitution.

STEAM STRIPPING

21. Steam stripping systems tested were effective at reducing the volatile organic concentration in aqueous waste streams and in recovering solvent product.

22. The effluent from the steam strippers evaluated at Facility G and M were discharged to wastewater treatment systems for final treatment. The effluent at Facility N was discharged to a river since the effluent meets National Pollutant Discharge Elimination System (NPDES) permit requirements.

23. Waste stream characteristics such as high concentrations of suspended solids can limit the removal efficiency of the steam stripper. Decanters are sometimes required upstream of the process to remove solids and any initial distinct organic layer prior to steam stripping.

CARBON ADSORPTION

24. Granular activated carbon (GAC) adsorption is effective at removing dissolved organic compounds from aqueous wastes containing low concentrations of organics after upstream processes have removed heavy metals and suspended solids.

25. The effluent from the GAC system at Facility B was discharged to the local municipal sewer system and at Facility C the effluent was sent to a nonhazardous surface impoundment on site for natural evaporation. The effluent stream from Facility C will be discharged to a creek when a NPDES discharge permit is obtained.

ACTIVATED SLUDGE

26. The activated sludge treatment system at Facility I removed greater than 90 percent of volatile organic compounds from an aqueous waste. Removals of Total Organic Carbon and Biochemical Oxygen Demand were greater than 85 and 94% respectively.

27. Metals were concentrated in the sludge; measurable quantities of zinc were also found in the effluent discharge.

SECTION 3

PROGRAM OVERVIEW

REGULATORY REVIEW

The EPA is proposing a framework for the regulation of land disposal of hazardous wastes. These regulations are required by the amendments to RCRA enacted through the Hazardous and Solid Waste Amendments of 1984 (HSWAs) on November 8, 1984. This action requires EPA to establish treatment standards for hazardous wastes according to schedules outlined in the amendments. Land disposal of hazardous wastes will be prohibited if these deadlines are not met, unless EPA determines such prohibition is not required to protect human health and the environment.

The amendments set statutory deadlines for the development of regulations for specific waste categories. EPA has published a proposed schedule for developing regulations according to these deadlines in the Federal Register (Vol. 50, No. 105, May 31, 1985, 23250). Regulations were established on November 7, 1986 for dioxin and solvent waste categories. The Best Demonstrated Available Technology (BDAT) standards for most spent solvent and dioxin wastes are based on high temperature incineration. Dilute solvent wastes (defined under the F001-F005 spent solvent rule as wastes having less than 1 percent total organic carbon) can be treated by steam stripping or biological treatment. However, dilute solvent wastes and all dioxin-containing wastes were exempted from treatment requirements for two years due to a significant shortage of treatment capacity. A full-scale field demonstration, using EPA's Mobile Incineration System operating at a 99.9999 percent destruction and removal efficiency (DRE) has demonstrated this technology for the treatment of dioxin-containing hazardous wastes. The Office of Research and Development has not addressed the treatment of dioxin wastes for this reason.

Effective July 8, 1987, HSWA prohibits disposal of hazardous wastes included in the California List. This list was developed by the California Department of Health Services for hazardous waste land disposal restrictions in California and has been included in HSWA by Congress. The wastes in the California list are a priority for the development of treatment standards and have been addressed by the Office of Research and Development program. Wastes included in the California list (RCRA Section 3001) are summarized below.

1. Liquid hazardous wastes containing free cyanides greater than or equal to 1,000 mg/l.

2. Liquid hazardous wastes containing the following metals or compounds thereof at concentrations greater than or equal to those specified.

   A. Arsenic - 500 mg/l

- B. Cadmium - 100 mg/l
- C. Chromium - 500 mg/l
- D. Lead - 500 mg/l
- E. Mercury - 20 mg/l
- F. Nickel - 134 mg/l
- G. Selenium - 20 mg/l
- H. Thallium - 130 mg/l

3. Liquid hazardous wastes with pH less than or equal to 2.0.

4. Liquid hazardous wastes containing polychlorinated biphenyls (PCBs) greater than or equal to 50 ppm.

5. Hazardous wastes containing halogenated organic compounds in total concentrations greater than or equal to 1,000 mg/kg. (RCRA sections 3004(d)(1) and (2), 42 U.S.C. 6924(d)(1) and (2).)

For the period ending November 8, 1988, contaminated soil and debris generated by the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA), Section 104 or 106, response actions and RCRA corrective actions are not subject to any land disposal prohibition or treatment standards for solvent, dioxin or California List wastes. Decisions regarding the deep well injection of solvent-laden wastes, dioxin-containing wastes or California List wastes must be made by August 8, 1988.

EPA must take action on all other listed hazardous wastes. The proposed schedule, described in the Federal Register, May 31, 1985, identifies other categories of hazardous wastes which must be evaluated by August 8, 1988, June 8, 1989 and May 8, 1990. Some of these hazardous wastes are also addressed in the California List. The wastes on the California List are based on specific concentrations present but integration with the Federal program requires EPA to address these listed wastes regardless of concentration. Hazardous wastes listed after November 8, 1984 must be evaluated for land disposal restrictions within six months of listing.

Land disposal includes any placement in landfill, surface impoundment, waste pile, injection well, land treatment, salt dome, salt bed, underground mine or cave. [RCRA Section 3004(k), 42 U.S.C.6924(k)]. Land disposal restrictions do not apply to wastes treated in surface impoundments as long as treatment residues are removed within one year [RCRA section 3005 (j)(11)(A)(B), 42 U.S.C 6925 (j)(11)(A)(B)] and the impoundment complies with design and location requirements specified in 40 CFR 265.221(a) through (e), 265 subpart F, 268.1(e)(2), 268.42 and 268.43. EPA may not find that a land disposal method is "protective" and exempt from regulation unless a petitioner demonstrates there will be no migration as long as the waste remains hazardous.

Regulations prohibiting land disposal are effective upon promulgation unless EPA sets another date because disposal or treatment capacity is limited until that date. Effective dates may not be extended beyond two years of the statutory deadline. Two one-year case-by-case extensions may be granted when an applicant demonstrates that, for reasons beyond his control, contractually committed capacity cannot be available by the effective date.

Management of hazardous wastes according to these regulations is dependent on both waste type and concentration. If wastes are not listed in 40 CFR, Part 268, subpart C, they are not subject to the land disposal restrictions. If wastes are listed, but constituent concentrations do not exceed levels listed in Table CCWE - Constitutent Concentration in Waste Extract under 268.41, the waste may be land disposed. If waste constituents exceed the specified levels, the waste is subject to the restrictions of Part 268. Treatment methods must be used if such technologies are specified for the waste in 268.40. If treatment methods are not specified, any appropriate method may be used to reduce constituent concentrations to levels below those specified in Table CCWE. The treated waste may be land disposed as long as concentrations are below those specified in Table CCWE.

SELECTION OF ORGANIC WASTES AND TREATMENT PROCESSES FOR EVALUATION

Solvents are one of the first hazardous waste categories to be addressed by HSWA. Evaluation of solvent and organic waste treatment processes is thus a priority of EPA and is a major focus of this study. Hazardous wastes addressed by the proposed Land Disposal Restrictions of Solvents are presented in Table 1. The November 7, 1986 final rule on Land Disposal Restrictions (FR Vol. 51, No. 216) addresses the F001 through F005 solvent wastes (including solvent mixtures) and have therefore, been addressed in this report. The following information on waste solvent generation, waste types generated (i.e., organic liquids, organic sludge, etc.) and available treatment technologies was obtained from the January 14, 1986 Federal Register - Proposed Treatment Standards for Hazardous Wastes Containing Solvents (Vol. 51, No. 9, 1711-1729). Approximately 3.1 billion gallons of waste solvents were generated in 1981. EPA estimates that 214 million gallons of solvent wastes, that are land disposed each year (including surface impoundments but excluding deep well injection), will require alternative disposal.

Liquids containing solvents of less than 1.0 percent organics and water (dilute aqueous liquid) comprise 185 million gallons of all solvent wastes land disposed. The majority, 99.9 percent, are disposed in surface impoundments. A total volume of 20.6 million gallons of organic liquids of more than 1.0 percent organics currently are land disposed. Of these, 10.9 million gallons contain principally halogenated organic constituents, and 9.7 million gallons contain nonhalogenated organics. Organic sludges and solids totalling 10.4 million gallons will require alternative disposal each year. Sludges and solids generally contain more than 1.0 percent organics and greater than 1.0 percent total solids. EPA assumes 7.2 million gallons of these materials contain primarily halogenated organic constituents and 3.2

TABLE 1. HAZARDOUS WASTES REGULATED BY LAND DISPOSAL RESTRICTIONS OF SOLVENTS

40 CFR 261.31	Hazardous Wastes
F001	Spent halogenated solvents from degreasing: tetrachloroethylene, trichloroethylene, methylene chloride, 1,1,1-trichloroethane, carbon tetrachloride and chlorinated flurocarbons.
F002	Spent halogenated solvents: tetrachloroethylene, methylene chloride, trichloroethylene, 1,1,1-trichloroethane, chlorobenzene, 1,1,2-trichloro-1,2,2-trifluoroethane, ortho-dichlorobenzene and trichlorofluoromethane.
F003	Spent nonhalogenated solvents: xylene, acetone, ethyl acetate, ethyl benzene, ethyl ether, methyl isobutyl ketone, n-butyl alcohol, cyclohexanone and methanol.
F004	Spent nonhalogenated solvents: cresols, cresylic acid and nitrobenzene.
F005	Spent nonhalogenated solvents: toluene, methyl ethyl ketone, carbon disulfide, isobutanol and pyridine.

40 CFR 261.33	Hazardous Wastes
P022	carbon disulfide
U002	acetone
U031	n butyl alcohol
U037	chlorobenzene
U052	cresols and cresylic acid
U057	cyclohexanone
U070	o-dichlorobenzene
U080	methylene chloride
U112	ethyl acetate

TABLE 1 (Continued). HAZARDOUS WASTES REGULATED BY LAND DISPOSAL RESTRICTIONS OF SOLVENTS

40 CFR 261.33	Hazardous Wastes
U117	ethyl ether
U121	trichorofluoromethane
U140	isobutanol
U154	methanol
U159	methyl ethyl ketone
U161	methyl isobutyl ketone
U169	nitrobenzene
U196	pyridine
U210	tetrachloroethylene
U211	carbon tetrachloride
U220	toluene
U226	1,1,1 trichloroethane
U228	trichloroethylene
U239	xylene

Solvent mixtures and still bottoms are also regulated.

General Definitions of Waste Categories

F - Hazardous wastes from non-specific process including spent solvents and process sludges FXXX wastes are identified as acutely hazardous wastes.

U & P - Wastes defined under RCRA as commercial chemical products, manufacturing chemical intermediates, off-specification species, container residues, and residues thereof

UXXX wastes are identified as toxic wastes
PXXX wastes are identified as acute hazardous wastes

million gallons contain nonhalogenated constituents. Additionally, 6.7 million gallons of inorganic sludges and solids contaminated with solvents require disposal. These wastes consist of contaminated soils, sludges and solids containing less than 1.0 percent organics. CERCLA wastes also require disposal, but estimates of these waste quantities are not yet available.

Steam stripping dilute volatile organics from water is demonstrated on wastes containing solvent concentrations up to several thousand milligrams per liter (mg/l). Recent information indicates that there are at least 27 industrial steam stripping units in operation. EPA is interested in developing data on steam stripping because it is applicable to the treatment of a wide variety of solvent-water mixtures. Steam stripping was hence chosen as one of the candidate alternative technologies for this study.

Activated carbon adsorption is a demonstrated technology for removal of organic compounds from water. Total organic constituents are normally less than a few hundred milligrams per liter. EPA has identified approximately 50 full scale commercial facilities utilizing carbon adsorption. Data describing this technology is required because it is applicable to a wide variety of solvent-water mixtures and it can remove organics to very low levels. When the treated wastewater approaches a predetermined level (breakthrough concentration) the spent carbon can either be regenerated and reused or destroyed in an incinerator. Carbon adsorption is often used in conjunction with other treatment methods, particularly as a final polishing step. Carbon adsorption was another technology chosen as a candidate alternative technology for this study.

Distillation is used to recover solvents and concentrated organics for reuse and combustion. EPA estimates there are at least 43 full scale commercial solvent recyclers using distillation to recover 149 million gallons of solvents per year. Data describing distillation is needed to determine the partitioning of trace contaminants between still bottoms and recovered distillate. Still bottoms need to be characterized because additional treatment is usually required. Both batch distillation and steam distillation processes were evaluated in this study.

Biological treatment involves the degradation of soluble organic compounds, through the use of microorganisms, in wastewaters when the organic concentrations in the wastewater are below levels which can be toxic to the biological organisms. Biological treatment processes are widely used, and a considerable amount of data exists documenting the biodegradation of soluble organic compounds. Biological treatment processes include aerobic treatment such as activated sludge systems, aerated lagoons, trickling filters, facultative degradation in waste stabilization ponds and anaerobic digestion. The activated sludge system is an aerobic process used to degrade organic compounds to carbon dioxide and water. Activated sludge treatment was evaluated at one facility during this study.

Incineration is used to destroy liquid wastes, containing solvents in low concentrations, and solids containing 60 percent or more solvents. At least 330 individual incineration units currently burn a minimum of 317 million gallons of materials containing solvents each year. Limited data are available characterizing air pollution control scrubber wastes. EPA is gathering additional data on the presence of hazardous constituents in incinerator ash and scrubber water. The two types of incinerators evaluated in this study are the rotary kiln and liquid injection incinerators.

An EPA survey documents that 1,400 industrial boilers burned chlorinated and nonchlorinated solvents in 1983. Results of test burns have shown that industrial boilers and furnaces can destroy 99.99 percent of difficult-to-burn organic constituents. Additional data is required to characterize commercial fuel blends. Waste blending for use as fuel was evaluated in this study.

Thin film evaporation is a demonstrated distillation process used to recover solvents for reuse and to produce a bottoms stream which is often used as fuel for incinerators. Three thin film evaporators were evaluated in this study.

SITE SELECTION PROCESS

Several approaches were considered appropriate for developing data on waste characteristics, treatment process performance, treatment residues and environmental emissions, which were required to supplement existing data. These include onsite pilot testing of selected industrial process waste streams; field evaluation of onsite (i.e. generator owned) treatment processes; and field evaluation of offsite (i.e. commercial) hazardous waste treatment facilities. The last approach was selected for the testing activities described here because it was considered to offer the best opportunity to quickly identify, evaluate and test existing processes treating hazardous wastes. Wastes are usually shipped offsite for disposal because onsite treatment either poses operational problems, or imposes excessive costs because of low waste volumes treated. Commercial facilities are capable of treating a wider variety of wastes with varying characteristics than many on-site facilities dedicated to a single waste stream.

Wastes shipped offsite have often been directed to land disposal in the past. Site screening was conducted to identify facilities that treat priority hazardous waste categories and wastes that have been directed to land disposal.

Selection and testing of treatment facilities consisted of:

1. Screening commercial hazardous waste treatment facilities through the use of the Industrial and Hazardous Management Firms Directory published by Environmental Information LTD., and telephone contacts to determine waste types treated, management practices, treatment processes, and willingness to participate in the program.

2. Site visits to selected facilities that are treating priority wastes.

3. Evaluation of site visit data to select sites for testing.

4. Performance of field sampling and process monitoring, following concurrence of EPA with the site selection, site specific Test Plan and Quality Assurance Project Plan.

SAMPLING PROGRAM

The general objectives of this sampling program are to collect and analyze samples from commercial hazardous waste TSDF's and to measure the performance of processes used at these facilities for treating or destroying hazardous wastes. The data resulting from this study are to be used to evaluate technologies for treatability of wastes that are being considered for the land disposal restrictions. Specific objectives were developed for each facility.

Sample locations at each site were selected to enable characterization of raw wastes, treated products, process residuals and emissions and to permit measurement of the performance of each unit operation. A composite liquid waste sampler (COLIWASA) was used to obtain samples from drums, tank trucks, and certain process vessels. The design and use of this type of sampling device is described in "Test Methods for Evaluating Solid Waste", SW-846, Section 1.2.1.1. Glass sampling bulbs were used to collect air samples as grabs.

Samples were split into fractions in the field and stored in appropriate containers. Preservatives were added for particular analyses, if practical. The samples were securely packed and forwarded to the analytical laboratory. No solvent, sludge or solid samples were chemically preserved. The analytical methods used in this sampling program are summarized in Table 2.

As part of this sampling program, certain samples were collected and analyzed to measure the precision and accuracy of the data. In addition, blank water samples were analyzed to identify any sampling or analytical contamination, such as from methylene chloride or acetone. The precision of the data can be expressed by the standard deviation of the analytical results of replicate samples. Accuracy or validity can be defined for a given sample matrix by measuring the percent recovery of spikes. Spikes are known concentrations of specific pollutants and surrogate compounds introduced into both the hazardous waste and blank water samples.

TABLE 2. ANALYTICAL AND SAMPLE PREPARATION METHODS AND REFERENCES

Parameter	Method Numbers (1)
1. Volatile Organics	Analytical Method 8240 Sample Preparation Method 5030
Chloromethane Bromomethane Vinyl Chloride Chloroethane Methylene chloride Acetone Carbon disulfide 1,1-dichloroethene 1,1-dichloroethane Trans-1,2-dichloroethene Chloroform 1,2-dichloroethene 2-Butanone 1,1,1-trichloroethane Carbon tetrachloride Vinyl acetate Bromodichloromethane 1,2-dichloropropane	Trans-1,3-dichloropropene Trichloroethene Dibromochloromethane 1,1,2-trichloroethane Benzene Cis-1, 3-dichloropropene 2-Chloroethylvinylether Bromoform 2-Hexanone 4-Methyl-2-pentanone Tetrachloroethene 1,1,2,2-Tetrachloroethane Toluene Chlorobenzene Ethylbenzene Styrene Total xylenes
2. Extractable Organics	Analytical Method 8270 Sample Preparation Method 3510
2,4,6-trichlorophenol p-chloro-m-cresol 2-chlorophenol 2,4-dichlorophenol 2,4-dimethylphenol 2-nitrophenol 4-nitrophenol 2,4-dinitrophenol 4,6-dinitro-o-cresol pentachlorophenol phenol acenaphthene benzidine 1,2,4-trichlorobenzene hexachlorobenzene hexachloroethane bis(2-chloroethyl)ether 2-chloronaphthalene 1,2-dichlorobenzene 1,3-dichlorobenzene 1,4-dichlorobenzene 3,3-dichlorobenzidene	bis(2-chloroethoxy)methane hexachlorobutadiene hexachlorocyclopentadiene isophorone naphthalene nitrobenzene N-nitrosodimethylamine N-nitrosodiphenylamine N-nitrosodi-n-propylamine bis(2-ethylhexyl)phthalate butyl benzyl phthalate di-n-butylphthalate di-n-octylphthalate diethylphthalate dimethyl phthalate benzo(a)anthracene benzo(a)pyrene 3,4-benzofluoranthene benzo(k)fluoranthene chrysene acenaphthylene anthracene

TABLE 2 (CONTINUED). ANALYTICAL AND SAMPLE PREPARATION METHODS AND REFERENCES

Parameter		Method Numbers (1)
	2,4-dinitrotoluene	benzo(ghi)perylene
	2,6-dinitrotoluene	fluorene
	1,2-diphenylhydrazine	phenanthrene
	fluoranthene	dibenzo(a,h)anthracene
	4-chlorophenyl phenyl ether	ideno(1,2,3,-cd)pyrene
	4-bromophenyl phenyl ether	pyrene
	bis(2-chloroispropyl)ether	2,3,7,8-tetrachlorodibenzo-p-dioxin
3.	Metals	
	Cadmium	Analytical Method 7130, Sample Prep. Method 3010
	Hexavalent Chromium	Analytical Method 218.5(2)
	Total Chromium	Analytical Method 7190 Sample Prep. Method 3010
	Copper	Analytical Method 7210 Sample Prep. Method 3010
	Lead	Analytical Method 7420 Sample Prep. Method 3010
	Nickel	Analytical Method 7520 Sample Prep. Method 3010
	Zinc	Analytical Method 7950 Sample Prep. Method 3010
4.	Chemical Oxygen Demand	Method 410.1(2)
5.	Total Organic Carbon	Method 415.2(2)
6.	Total Organic Halide	Method 9020
7.	Oil and grease	Method 310.1(2)
8.	Residue	
	Total Solids	Method 160.3(2)
	Total Volatile Solids	Method 160.4(2)
	Total Suspended Solids	Method 160.2(2)
	Total Dissolved Solids	Method 160.1(2)
	Total Volatile Suspended Solids	Method 160.4(2)
	Total Volatile Dissolved Solids	Method 160.4(2)
9.	Cyanide - Total and Amenable	Method 9010
10.	Ammonia	Method 350.2(2)
11.	Sulfide	Method 9030

TABLE 2 (CONTINUED). ANALYTICAL AND SAMPLE PREPARATION METHODS AND REFERENCES

Parameter	Method Numbers (1)
12. Chloride	Qualitative
13. Sulfate	Qualitative
14. Fluoride	Method 340.1(2)
15. Silica	Method 370.1(2)
16. Acidity	Method 305.1(2)
17. Alkalinity	Method 310.1(2)
18. EP Toxicity:	Sample Prep. Method 1310
Metals	
Arsenic	Analytical Method 7061, Sample Prep. Method 3010
Barium	Analytical Method 7080, Sample Prep. Method 3010
Cadmium	Analytical Method 7130, Sample Prep. Method 3010
Chromium	Analytical Method 7190, Sample Prep. Method 3010
Lead	Analytical Method 7420, Sample Prep. Method 3010
Mercury	Analytical Method 7470, Sample Prep. Method 3010
Selenium	Analytical Method 7741, Sample Prep. Method 3010
Silver	Analytical Method 7760, Sample Prep. Method 3010
19. TCLP:	Sample Prep. Method 13xx(3)
Metals	
Arsenic	Analytical Method 7061, Sample Prep. Method 3010

TABLE 2 (CONTINUED). ANALYTICAL AND SAMPLE PREPARATION METHODS AND REFERENCES

	Parameter	Method Numbers (1)
	Barium	Analytical Method 7080, Sample Prep. Method 3010
	Cadmium	Analytical Method 7130, Sample Prep. Method 3010
	Chromium	Analytical Method 7190, Sample Prep. Method 3010
	Lead	Analytical Method 7420, Sample Prep. Method 3010
	Mercury	Analytical Method 7470, Sample Prep. Method 3010
	Selenium	Analytical Method 7741, Sample Prep. Method 3010
	Silver	Analytical Method 7760, Sample Prep. Method 3010
	Volatile Organics	Analytical Method 8240, Sample Prep. Method 5030
	TCLP extracts were analyzed for the same 35 volatile organics compounds which are listed as item 1 of Table 4.	
20.	Percent Carbon	ASTM D3178(4)
21.	Percent Hydrogen	ASTM D3178(4)
22.	Percent Water	ASTM D1364(4)
23.	BTU Content	ASTM D2015(4)

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1. Reference is Test Methods for Evaluating Solid Wastes, SW-846, 2nd edition, U.S. EPA, July 1982, unless otherwise specified.

2. Method for Chemical Analysis of Water and Waste, EPA-600/4-79-020, March 1983.

3. "Toxicity Characteristic Leaching Procedure", Federal Register, Vol. 51, No. 9, January 14, 1986. pp 1750-1758.

4. 1984 Annual Book of ASTM Standards

SECTION 4

SELECTED ALTERNATIVE TREATMENT TECHNOLOGIES FOR ORGANIC HAZARDOUS WASTES

The objective of this project is to measure the performance of specific technologies to treat hazardous organic waste streams. Table 3 is a matrix of the treatment technologies and waste streams that were studied and identification codes for facilities at which the sampling took place. The treatment technologies discussed in sections 5 through 12 of this report include batch distillation, steam distillation, steam stripping, agitated thin film evaporation, heated screw auger evaporation, incineration, waste blending/ feed substitution, activated sludge treatment, and activated carbon treatment. The waste streams have been broken down into four categories: dilute aqueous liquid, pumpable organic sludge, nonpumpable solid waste and organic liquids. For the purpose of this project, dilute aqueous liquids are defined as wastewaters which contain less than 1 percent organics. Organic liquids on the other hand are composed of greater than 50 percent organics. Pumpable organic sludges, such as petroleum refinery sludges, contain greater than 1 percent solids and 10 percent organics. Nonpumpable solid wastes are designated as such because of their viscous nature not the organic content of the waste therefore no designation of percentage of organics was specified. The quantities of these wastes generated has been discussed previously in Section 3.

TABLE 3. TREATMENT TECHNOLOGY/WASTE STREAM/FACILITY MATRIX

Waste / Process	Dilute Aqueous Liquid (<1% Organics)	Pumpable Organic Sludge (>1% Solids) (>10% Organics)	Non – Pumpable Solid Waste	Organic Liquid (>50% Organics)
Batch Distillation				A,D
Steam Distillation				C
Steam Stripping	G,M,N			
Agitated Thin Film Evaporation		J,K,L		
Heated Screw Auger Evaporation			K	
Incineration			F,H	F,H
Waste Blending/ Fuel Substitution		B	B	B
Activated Sludge Treatment	I			
Activated Carbon Treatment	B,C			

A – D and F – N are Facilities sampled by Metcalf & Eddy, Alliance and RTI for which results are summarized in this report. There is no facility E designated in this report.

SECTION 5

BATCH DISTILLATION

Distillation is used extensively for the reclamation of usable solvent from spent or contaminated solvent wastes. Two distillation processes were included in the sampling program. These were the simple batch distillation process at Facility A and the series batch distillation processes at Facility D.

THEORY

Distillation is a physical process in which miscible liquids are separated on the basis of their different volatilities. Figure 1 represents a typical batch distillation unit. In simple batch distillation (differential distillation), the feed is charged to a still pot and heated indirectly with steam or oil in an internal coil or external jacket. The boiling point of the mixture is that temperature at which the sum of the vapor pressures exerted by the individual components is equal to the system pressure. The partial pressure contributed by each component is a function of the vapor pressure that would be exhibited by the pure compound at that temperature and the mole fraction of that compound in the liquid mixture. The vapor above the boiling liquid is enriched in the most volatile or lowest boiling component, while the liquid is enriched in the less volatile components. Vapors rising from the still pot are condensed, typically by heat exchange with non-contact cooling water, and the condensed distillate is collected in a receiver. As distillation of the batch proceeds, the temperature of the boiling liquid in the still pot increases and the composition of the overhead product, or distillate, changes gradually, becoming leaner in the most volatile component and richer in less volatile components. By collecting distillate in a series of separate receivers, distinct product cuts can be recovered.

For example, given a ternary mixture of organic liquids containing a small amount of a volatile component A, a majority of component B of intermediate volatility, and a small amount of component C of a very low volatility, two product cuts might be collected. The initial product cut would contain mostly component A. A large volume second cut would be rich in component B. The residual material in the still pot (still bottoms) would be largely component C. While all three fractions would contain some concentration of each component, a degree of separation would be achieved.

The simple batch still provides only one equilibrium stage or "theoretical plate" of separation. Better separation and purer product cuts can be achieved by adding a rectifying column (packed or tray tower) between the still pot and condenser and by refluxing a fraction of the condensed distillate back through the column, as shown in Figure 1.

Batch distillation is a useful technology for the treatment of spent solvent wastes. Volatile solvent can be separated from nonvolatile

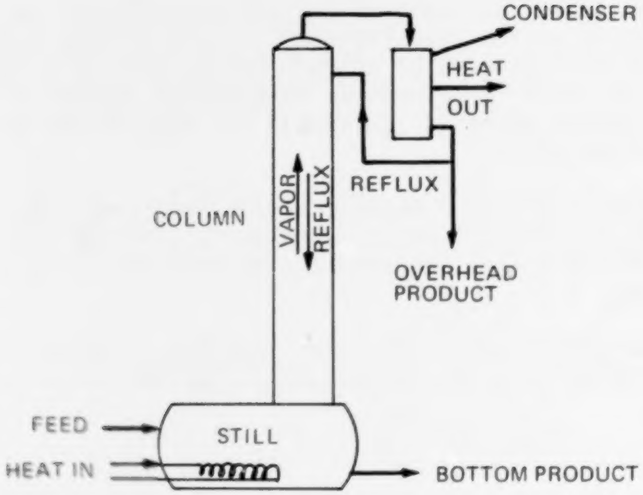
CONDENSER
HEAT
OUT
REFLUX
COLUMN
VAPOR
REFLUX
OVERHEAD
PRODUCT
FEED
STILL
HEAT IN
BOTTOM PRODUCT

Figure 1. Batch Distillation Schematic.

(McCabe, W.L. and Smith, J.C., "Unit Operations of Chemical Engineering", 3rd Ed., McGraw Hill, Inc., 1976) impurities such as oil, grease, inorganic solids, and polymeric resins. In general, simple batch distillation is appropriate when a crude separation is acceptable and the wastes are highly concentrated and yield significant amounts of recoverable solvent.

Batch distillation processes generate a residue referred to as "still bottoms" which contains the nonvolatile impurities of the original waste and may also contain significant amounts of solvent. The physical and chemical nature of the still bottoms and its ultimate disposition depend on the composition of the original waste, the temperature of the distillation and the batch process time. Still bottoms resulting from the distillation of typical kinds of spent solvent wastes include:

1. A free-flowing oil/solvent mixture suitable for use as a fuel substitute or for fuel blending. These bottoms result from treatment of non halogenated solvent wastes such as toluene and methyl ethyl ketone or treatment of halogenated solvent wastes to a chlorine content which is low enough for the bottoms to be used as a fuel substitute.

2. A highly chlorinated organic sludge resulting from treatment of a halogenated solvent waste, such as 1,1,1-trichloroethane, which must be solidified, drummed and shipped to a secure landfill for disposal.

3. An inorganic sludge containing lead and chromium paint pigments which might require chemical stabilization prior to landfill disposal.

FACILITY A

Process Description

Facility A is the only treatment plant owned by Company A, a privately-owned hazardous waste treatment firm formed in 1981. The batch distillation process for solvent reclamation has been in operation at Facility A since 1983. Solvents recovered by this process are sold to various customers for use as degreasers, glass cleaners, paint or lacquer thinners. Organic liquid still bottoms are sold as fuel to oil reclaimers, power companies and cement kiln operators.

Facility A accepts and processes both chlorinated and nonchlorinated solvent wastes, including the following types:

Chlorinated

Principal Constituents:

methylene chloride
1,1,1 trichloroethane
freons

Non-chlorinated

Principal Constituents:

Ketones

acetone
methyl ethyl ketone

Alcohols

methanol
isopropanol

Aromatics

toluene
xylenes (ortho and para)

Facility A prefers waste solvents containing greater than 60 percent of the major solvent component. Wastes containing less than 40 percent of the major solvent component are not accepted due to unfavorable economics for reclamation. The upper limit on the suspended solids content of waste solvents is about 10 to 15 percent. Wastes with higher solids content may be accepted for processing, but the generator is charged a premium for disposal of the resulting sludge.

The waste solvents processed at Facility A come from a variety of generators. The electronics industry is the major industry served. Other generators include fiberglass manufacturers, aluminum fabricators, casket manufacturers, and automobile service garages. Due to their original uses, the waste solvents typically contain oils and grease, paint solids, plastics, dirt and grit as the major contaminants.

Two types of spent halogenated solvents (methylene chloride - F001, and a mixture of isopropanol, freon and 1,1,1-trichloroethane - F002) and two spent non-halogenated solvents (methyl ethyl ketone - F005, and isopropanol - D001) were treated by batch distillation during the four day test program.

Figure 2 is a simple flow diagram for the batch distillation process at Facility A. Two parallel distillation systems share a common feed pump and filter. The two systems differ only slightly in condenser design and process instrumentation.

A centrifugal pump is used to pump waste solvents from drums into one of the two 500-gallon Pfaudler glass-lined still pots. An electric mixer is used to agitate drums with settled solids before pumping. Solvents are pumped through a metal screen (approximately 1/8-inch diameter openings) and a bag-type fabric filter (400 mesh) to remove any suspended solids.

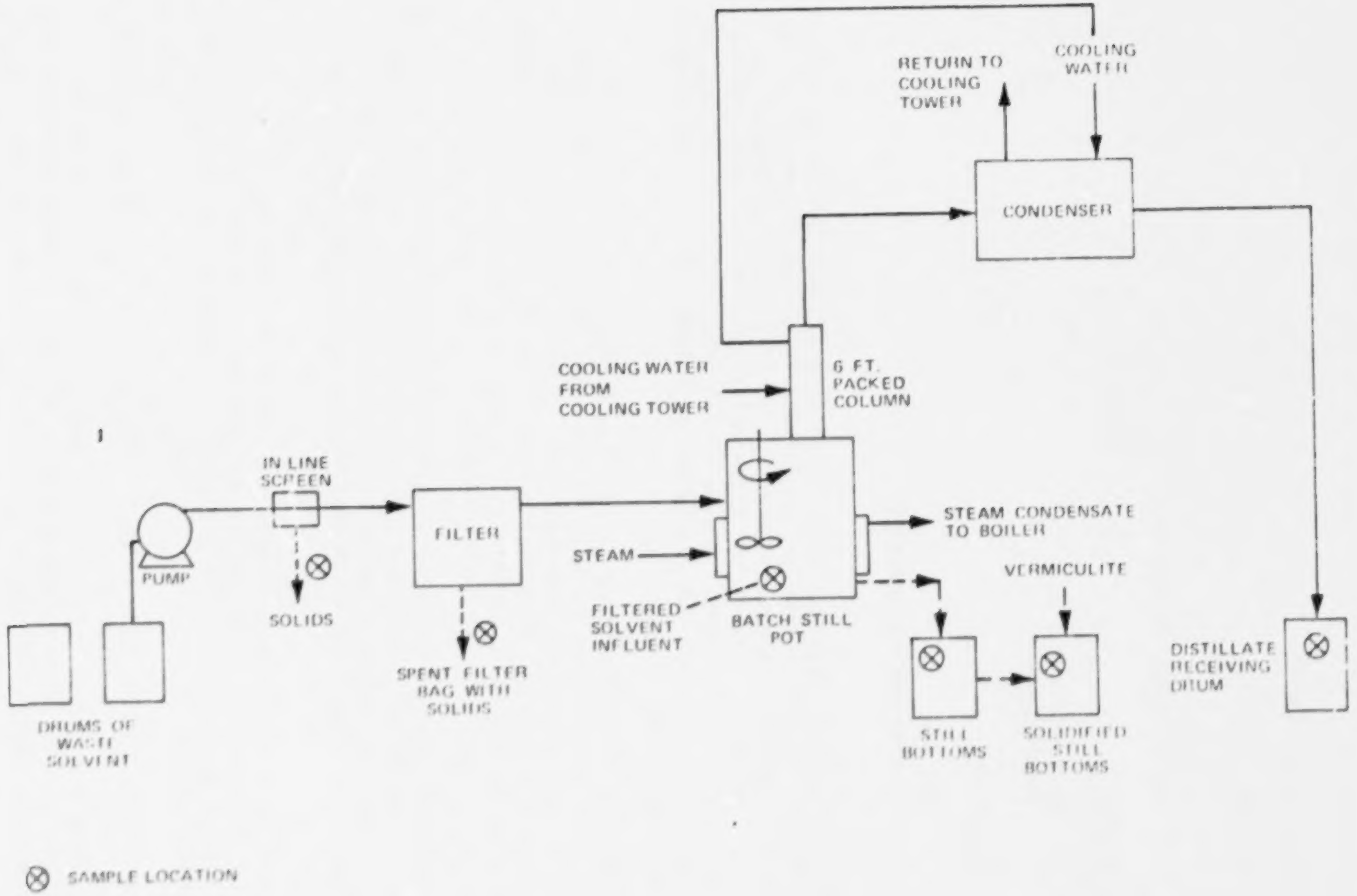
RETURN TO COOLING TOWER
COOLING WATER
CONDENSER
COOLING WATER FROM COOLING TOWER
6 FT. PACKED COLUMN
IN LINE SCREEN
FILTER
STEAM
STEAM CONDENSATE TO BOILER
VERMICULITE
PUMP
SOLIDS
FILTERED SOLVENT INFLUENT
BATCH STILL POT
DISTILLATE RECEIVING DRUM
SPENT FILTER BAG WITH SOLIDS
STILL BOTTOMS
SOLIDIFIED STILL BOTTOMS
DRUMS OF WASTE SOLVENT
⊗ SAMPLE LOCATION

Figure 2. Flow diagram/sample points for batch distillation at Facility A.

After all drums (usually eight 55-gallon drums) for the batch have been emptied, the Pfaudler reactor's constant-speed, 75 RPM agitator is started to homogenize the liquid. A plant operator uses a polyethylene "thief" (a 2-1/2-foot long, 3/4-inch diameter hollow tube sampler) to grab a sample of this mixed waste solvent from the still pot. After sampling, the still pot's access hatch is bolted closed. Steam is then applied to heat the jacketed still pot.

Both reactors have a 6-foot tall packed column which provides some fractionation. The column is jacketed to allow cooling with water from the facility's cooling tower. The solvent waste is heated to above its boiling point in the still pot while the column is cooled to the boiling point of the desired solvent product thus effectively providing some refluxing of the solvent vapors back to the still pot.

Overhead vapors which rise above the packed column are condensed in a water-cooled shell and tube heat exchanger. The liquid distillate flows from this condenser through a 2-inch diameter pipe into a clean 55-gallon drum. The pipe extends into a 10-inch diameter screened funnel inserted into one 2-inch diameter bung hole of the drum. The second bung hole is kept open so that the drum is vented to the atmosphere during filling. When the drum is nearly full or when a rise in the column temperature indicates that the composition of the distillate has changed, a valve in the distillate pipe above the drum is manually closed, the drum is removed and an empty drum is put in its place. The valve is then reopened so that distillate flows to the new receiving drum.

After all recoverable product is collected, as indicated by cessation of distillate flow or by column temperature rise above the desired product boiling point, steam flow to the still pot jacket is shut off. After the steam is shut off, a valve at the bottom of the still pot is opened to allow the hot still bottoms to drain out into an open cart or a drum. If necessary, an L-shaped piece of pipe is inserted manually through the valve to dislodge any solid material which clogs the discharge. Depending on their pumpability, flammability, chlorine content, and BTU content, still bottoms may be blended and sold as "secondary fuel" or solidified with vermiculite and sent to a hazardous waste landfill.

Assembling drums and filling the still pot took two to three hours. During the distillation process, a single operator was dedicated to monitoring a batch. Thirty to 60 minutes were required to preheat the still pot before distillation commenced. Distillation typically required six or more hours.

The distillation process at Facility A is currently limited by the available steam temperature from the boiler. Solvents with a boiling point greater than 140°C cannot be distilled. Future addition of a vacuum system to one batch still and a larger boiler was planned to allow distillation of higher temperature boiling solvents and increase processing capacity.

The principal goals of the distillation process at Facility A are:

1. To recover recyclable solvent from waste solvents. Facility A does not attempt to fractionate solvents to high purity specifications; rather, they make a rough cut to separate clean, clear solvent from nonvolatile components like oils, greases, dirt and paint solids. The distillate cuts are sold for their solvent value in noncritical products like lacquer thinner, degreaser, and glass cleaner.

2. To produce a pumpable, high BTU-content, low chlorine still bottoms, which can be blended into secondary fuels for sale to oil reclaimers, power plants or cement kilns.

3. To minimize the volume of solvent waste residuals that cannot be recycled for solvent or fuel value and must be properly disposed.

Sampling Program

The onsite sampling program at Facility A took place from June 24, 1985 through June 28, 1985. Four batches of waste solvents were distilled during the sampling episode. Samples were collected from the following locations to evaluate each distillation batch:

- filtered solvent influent;
- distillate receiving drum;
- spent filter bag with solids;
- spent filter solids from in-line screen;
- still bottoms.

The sampling points are identified on the process flow diagram of Figure 2 and are summarized in Table 4.

Data

Waste Characterization --

The generator's "UNIFORM HAZARDOUS WASTE MANIFEST" for all solvent wastes distilled at Facility A during June 25 through 27, 1985 were obtained. Table 5 summarizes the information contained on these forms. Because Facility A accepts small quantities of waste solvent from small volume generators, some batches of solvents for distillation are made up of drums from a number of sources. This was the case for the material distilled as Batch 4. Analytical data for the samples taken of the batch distillation process are summarized in Tables 6-9.

The filtered solvent influent of batch 1 (Table 6) was a mixture of three major components - 1,1,1 trichloroethane (740,000 mg/l), freon 113 (570,000 mg/l) and isopropanol (190,000 mg/l) - with smaller concentrations of acetone, dioxane, and ethanol. The water content was just under one percent. Organic chloride made up 54 percent of the total mass. The

TABLE 4. FACILITY A SAMPLE POINTS AND SAMPLING METHODS

Sample Reference	Description	Type of Sample	Time Interval
BATCH DISTILLATION			
Filtered Solvent Influent	After the drums of waste solvent had been pumped into the still pot and the agitator had been started to mix the liquid, a single grab sample was collected from the still pot via a glass COLIWASA.	Grab	One per treatment cycle
Distillate Cut	Each sample is collected directly from the distillate receiving drum using a glass COLIWASA, after that drum has been taken off-line.	Grab	Four per treat-ment sample, from the first, third, fifth and seventh distillate receiving drums.
Spend Filter Solids-Bag	In-line strainer filter bag was collected at the conclusion of filtration for solvent batches 3 and 4.	Grab	One per treatment cycle
Spent Filter Solids-Screen	A sample of solids flushed out of the in-line screen into a collection pan was taken from the pan using a beaker or trowel at the conclusion of filtration for solvent batches 1 and 3.	Grab	One per treatment cycle
Still Bottoms	Samples were collected from the still bottoms receiving drums or cart after all material had drained from the still pot after the distillation run. A glass beaker or COLIWASA was used.	Grab	Once per treatment cycle
Solidified Still Bottoms	No samples were collected by M&E personnel during the sampling trip, because still bottoms were not solidified during the week of June 24.	Grab	Once per treatment cycle, if still bottoms are landfilled.

TABLE 5. GENERATOR'S MANIFEST DATA FOR WASTE SOLVENTS

Day	Batch	Generator Industry Type	Major Components	Waste Code
1	1	Electronics	1,1,1-trichloroethane freon 113 isopropanol	F001
2	2	Electronics	isopropanol	D001
3	3	Biotechnology Research	methylene chloride	F001
3	4	Electronics Plating Specialty Chemical Manufacturer Motor Rebuilder	methyl ethyl ketone	F005

TABLE 6. BATCH DISTILLATION ANALYTICAL DATA-BATCH 1 FACILITY A

PARAMETER	UNITS	FILTERED SOLVENT INFLUENT 10004	FILTERED SOLIDS-SCREEN 10005	STILL BOTTOMS 10006	PRODUCT CUT 1 10007	PRODUCT CUT 2 10008	PRODUCT CUT 3 10009	PRODUCT CUT 4 10010
VISCOSITY(@25 C)	cSt	NM	NM	4.55	NM	NM	NM	NM
PAINT FILTER TEST	--	NM	NM	FAIL	NM	NM	NM	NM
DENSITY	G/ML	1.2	NS	0.952	1.32	1.24	1.2	1.195
COD	MG/L	NS	NS	I	NS	NS	NS	NS
TOTAL SOLIDS	WT %	3.0	4.9	71	NS	NS	NS	NS
TOTAL VOLATILE SOLIDS	% of TS	100	NS	100	NS	NS	NS	NS
TOTAL SUSPENDED SOLIDS	MG/L	460	NS	5700	NS	NS	NS	NS
TOTAL VOL SUS SOLIDS	MG/L	440	NS	5600	NS	NS	NS	NS
TOTAL VOL DIS SOLIDS	MG/L	6100	NS	560000	NS	NS	NS	NS
TOTAL DISSOLVED SOLIDS	MG/L	6200	NS	560000	NS	NS	NS	NS
SILICA	WT %	NA	0.68	NA	NS	NS	NS	NS
METALS								
COPPER	MG/KG	NA	3.4	NA	NS	NS	NS	NS
CHROMIUM	MG/KG	NA	0.47	NA	NS	NS	NS	NS
SILVER	MG/KG	NA	0.2	NA	NS	NS	NS	NS
IRON	MG/KG	NA	4.2	NA	NS	NS	NS	NS
POC								
ACETONE	MG/L	2300	NS	<5000	3100	9800	3700	2800
BUTANOL	MG/L	<500	NS	<5000	<500	<500	<500	<500
ISO-BUTANOL	MG/L	<500	NS	<5000	<500	<500	<500	<500
DIOXANE	MG/L	33000	NS	150000	12000	5600	15000	97000
ETHANOL	MG/L	11000	NS	<5000	18000	11000	9200	3900
FREON 113	MG/L	570000	NS	<5000	640000	390000	140000	<500
ISOPROPANOL	MG/L	190000	NS	<5000	210000	130000	290000	260000
METHANOL	MG/L	<500	NS	<5000	<500	<500	<500	<500
METHYLENE CHLORIDE	MG/L	<500	NS	<5000	<500	<500	<500	<500
METHYL ETHYL KETONE	MG/L	<500	NS	<5000	<500	<500	<500	<500
1,1,1-TRICHLOROETHANE	MG/L	740000	NS	370000	460000	430000	680000	340000
TOLUENE	MG/L	T <500	NS	<5000	<500	<500	<500	<500
P-XYLENE,M-XYLENE	MG/L	<500	NS	<5000	<500	<500	<500	<500
O-XYLENE	MG/L	<500	NS	<5000	<500	<500	<500	<500
TOTAL ORGANIC HALIDE	WT % Cl	53.17	NS	8.85	29.79	53.33	61.4	60.47
EP TOX		NS	NS	PASS	NS	NS	NS	NS
ARSENIC	MG/L	NS	NS	<0.1	NS	NS	NS	NS
BARIUM	MG/L	NS	NS	<0.10	NS	NS	NS	NS
CADMIUM	MG/L	NS	NS	0.031	NS	NS	NS	NS
CHROMIUM	MG/L	NS	NS	<0.05	NS	NS	NS	NS
LEAD	MG/L	NS	NS	0.19	NS	NS	NS	NS
MERCURY	MG/L	NS	NS	<0.1	NS	NS	NS	NS
SELENIUM	MG/L	NS	NS	<1	NS	NS	NS	NS
SILVER	MG/L	NS	NS	<0.2	NS	NS	NS	NS
TCLP		NS	NS	#	NS	NS	NS	NS
FLASHPOINT	F	<75	NS	NS	NS	NS	NS	NS
BOILING RANGE	C	60-81	NS	NS	NS	NS	NS	NS
% CARBON	WT %	28.12	20.62	73.33	22.19	23.56	25.63	27.3
% HYDROGEN	WT %	4.85	2.91	11.82	3.19	3.94	4.26	4.65
% WATER	WT %	0.93	1.17	0.04	1.49	1.48	0.57	0.08
BTU CONTENT	BTU/LB	NS	NS	15722	NS	NS	NS	NS

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FOR PRINCIPLE ORGANIC COMPOUNDS, POLLUTANTS NOT LISTED WERE NOT DETECTED IN ANY OF THE SAMPLES ABOVE THE ANALYTICAL DETECTION LIMIT.

TABLE 7. BATCH DISTILLATION ANALYTICAL DATA-BATCH 2 FACILITY A

PARAMETER	UNITS	FILTERED SOLVENT INFLUENT 10015	STILL BOTTOMS 10017	PRODUCT CUT 1 10018	PRODUCT CUT 2 10019	PRODUCT CUT 3 10020	PRODUCT CUT 4 10021
VISCOSITY		NM	NM	NM	NM	NM	NM
PAINT FILTER TEST	--	NM	FAIL	NM	NM	NM	NM
DENSITY	G/ML	0.834	0.789	0.979	0.816	0.81	0.812
COD	MG/L	NS	I	NS	NS	NS	NS
TOTAL SOLIDS	WT %	0.16	10.0	NS	NS	NS	NS
TOTAL VOLATILE SOLIDS	% of TS	98	100	NS	NS	NS	NS
TOTAL SUSPENDED SOLIDS	MG/L	8.4	14	NS	NS	NS	NS
TOTAL VOL SUS SOLIDS	MG/L	5	12	NS	NS	NS	NS
TOTAL VOL DIS SOLIDS	MG/L	1600	100000	NS	NS	NS	NS
TOTAL DISSOLVED SOLIDS	MG/L	1600	100000	NS	NS	NS	NS
SILICA	WT %	< 0.05	NA	NS	NS	NS	NS
METALS							

COPPER	MG/KG	2.9	NA	NS	NS	NS	NS
CHROMIUM	MG/KG	0.28	NA	NS	NS	NS	NS
SILVER	MG/KG	< .1	NA	NS	NS	NS	NS
IRON	MG/KG	6.5	NA	NS	NS	NS	NS
POC							

ACETONE	MG/L	<500	<500	<5	<500	<500	<500
BUTANOL	MG/L	<500	<500	<5	<500	<500	<500
ISO-BUTANOL	MG/L	<500	<500	<5	<500	<500	<500
DIOXANE	MG/L	<500	<500	<5	<500	<500	<500
ETHANOL	MG/L	3800	2100	3100	3300	3100	3200
ISOPROPANOL	MG/L	690000	350000	400000	410000	510000	700000
METHANOL	MG/L	<500	<500	<5	<500	<500	<500
METHYLENE CHLORIDE	MG/L	<500	<500	<5	<500	<500	<500
METHYL ETHYL KETONE	MG/L	<500	<500	<5	<500	<500	<500
TOLUENE	MG/L	<500	<500	<5	<500	<500	<500
P-XYLENE,M-XYLENE	MG/L	<500	<500	<5	<500	<500	<500
O-XYLENE	MG/L	740	10000	<5	T <500	T <500	720
TOTAL ORGANIC HALIDE	WT % Cl	0.032	0.071	0.57	<0.5	<0.6	0.032
EP TOX		NS	FAIL	NS	NS	NS	NS

ARSENIC	MG/L	NS	<0.1	NS	NS	NS	NS
BARIUM	MG/L	NS	<0.10	NS	NS	NS	NS
CADMIUM	MG/L	NS	0.094	NS	NS	NS	NS
CHROMIUM	MG/L	NS	0.31	NS	NS	NS	NS
LEAD	MG/L	NS	1) 8.2	NS	NS	NS	NS
MERCURY	MG/L	NS	<0.1	NS	NS	NS	NS
SELENIUM	MG/L	NS	<1	NS	NS	NS	NS
SILVER	MG/L	NS	<0.2	NS	NS	NS	NS
FLASHPOINT	F	<75	NS	NS	NS	NS	NS
BOILING RANGE	C	73-93	NS	NS	NS	NS	NS
% CARBON	WT %	54.61	65.73	49.01	54.28	55.22	57.61
% HYDROGEN	WT %	12.72	13.98	11.44	13.26	13.66	13.49
% WATER	WT %	3.66	2.86	4.8	5.68	7.53	4.96
BTU CONTENT	BTU/LB	NS	15244	NS	NS	NS	NS

FOOTNOTES
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1) EXCEEDED RANGE OF CALIBRATION CURVE, HOWEVER, FORMED AN OILY LAYER UPON DILUTION WHICH GAVE NO ABSORBANCE.

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TABLE 8. BATCH DISTILLATION ANALYTICAL DATA-BATCH 3 FACILITY A

PARAMETER	UNITS	FILTERED SOLVENT INFLUENT 10032	FILTERED SOLIDS-BAG 10033	STILL BOTTOMS 10034	PRODUCT CUT 1 10035	PRODUCT CUT 2 10036	PRODUCT CUT 3 10037	PRODUCT CUT 4 10038
VISCOSITY		NM	NM	NM	NM	NM	NM	NM
PAINT FILTER TEST	--	NM	NM	FAIL	NM	KM	NM	NM
DENSITY	G/ML	1.03	NM	0.856	1.24	1.18	0.953	0.852
COD	MG/L	NS	NS	I	NS	NS	NS	NS
TOTAL SOLIDS	WT %	0.069	39	0.63	NS	NS	NS	NS
TOTAL VOLATILE SOLIDS	% of TS	90	66	82	NS	NS	NS	NS
TOTAL SUSPENDED SOLIDS	MG/L	5)	NS	170	NS	NS	NS	NS
TOTAL VOL SUS SOLIDS	MG/L	5)	NS	160	NS	NS	NS	NS
TOTAL VOL DIS SOLIDS	MG/L	550	NS	4100	NS	NS	NS	NS
TOTAL DISSOLVED SOLIDS	MG/L	620	NS	4800	NS	NS	NS	NS
SILICA	WT %	<0.004	6.82	<0.005	NS	NS	NS	NS
METALS								
COPPER	MG/KG	0.97	0.21	2.3	NS	NS	NS	NS
CHROMIUM	MG/KG	<.025	4.3	0.096	NS	NS	NS	NS
SILVER	MG/KG	<.05	0.4	<.05	NS	NS	NS	NS
IRON	MG/KG	0.45	11200	1.4	NS	NS	NS	NS
POC								
ACETONE	MG/L	9500	NS	1300	4500	5600	11000	12000
BUTANOL	MG/L	1800	NS	7500	<500	<500	770	T <500
ISO-BUTANOL	MG/L	2300	NS	640	2900	3100	12000	49000
DIOXANE	MG/L	1700	NS	4000	<500	<500	2600	2800
ETHANOL	MG/L	14000	NS	14000	4200	<500	990	17000
ISOPROPANOL	MG/L	37000	NS	98000	8100	8800	37000	72000
METHANOL	MG/L	300000	NS	230000	160000	190000	180000	190000
METHYLENE CHLORIDE	MG/L	460000	NS	<500	1100000	710000	590000	58000
METHYL ETHYL KETONE	MG/L	240000	NS	370000	42000	65000	280000	340000
TOLUENE	MG/L	46000	NS	110000	7300	17000	290000	350000
P-XYLENE,M-XYLENE	MG/L	T <500	NS	3100	<500	<500	5600	8900
O-XYLENE	MG/L	<500	NS	T <500	<500	<500	T <500	570
TOTAL ORGANIC HALIDE	WT % Cl	41.58	NS	1.07	66.72	69.25	7.76	4.72
EP TOX		NS	NS	PASS	NS	NS	NS	NS
ARSENIC	MG/L	NS	NS	<0.1	NS	NS	NS	NS
BARIUM	MG/L	NS	NS	<1	NS	NS	NS	NS
CADMIUM	MG/L	NS	NS	<0.4	NS	NS	NS	NS
CHROMIUM	MG/L	NS	NS	<0.3	NS	NS	NS	NS
LEAD	MG/L	NS	NS	<0.5	NS	NS	NS	NS
MERCURY	MG/L	NS	NS	<0.1	NS	NS	NS	NS
SELENIUM	MG/L	NS	NS	<1	NS	NS	NS	NS
SILVER	MG/L	NS	NS	<0.2	NS	NS	NS	NS
TCLP		NS	NS	#	NS	NS	NS	NS
FLASHPOINT	F	<75	NS	NS	NS	NS	NS	NS
BOILING RANGE	C	44-85	NS	NS	NS	NS	NS	NS
% CARBON	WT %	40.00	85.47	57.81	22.12	24.84	45.78	56.18
% HYDROGEN	WT %	7.94	13.79	11.41	5.13	5.42	9.08	10.5
% WATER	WT %	3.59	6.00	7.13	1.59	2.01	3.81	5.04
BTU CONTENT	BTU/LB	NS	NS	12339	NS	NS	NS	NS

FOOTNOTES

5) FILTERS DISSOLVED UNDER VACUUM

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TABLE 9. BATCH DISTILLATION ANALYTICAL DATA-BATCH 4 FACILITY A

PARAMETER	UNITS	FILTERED SOLVENT INFLUENT 10044	STILL BOTTOMS 10046	PRODUCT SLOP CUT 1 10047	PRODUCT SLOP CUT 2 10048	PRODUCT SLOP CUT 3 10049	PRODUCT SLOP CUT 4 10050
VISCOSITY		NM	NM	NM	NM	NM	NM
PAINT FILTER TEST	--	NM	FAIL	NM	NM	NM	NM
DENSITY	G/ML	0.941	0.858	1.07	0.996	0.913	0.845
COD	MG/L	NS	I	NS	NS	NS	NS
TOTAL SOLIDS	WT %	1) 4900	6.7	NS	NS	NS	NS
TOTAL VOLATILE SOLIDS	% of TS	1) 4900	98	NS	NS	NS	NS
TOTAL SUSPENDED SOLIDS	MG/L	110	1400	NS	NS	NS	NS
TOTAL VOL SUS SOLIDS	MG/L	37	860	NS	NS	NS	NS
TOTAL VOL DIS SOLIDS	MG/L	4800	66000	NS	NS	NS	NS
TOTAL DISSOLVED SOLIDS	MG/L	4900	66000	NS	NS	NS	NS
SILICA	WT %	NA	0.01	NS	NS	NS	NS
METALS							
COPPER	MG/KG	1) 0.47	2.5	NS	NS	NS	NS
CHROMIUM	MG/KG	1) 0.44	0.65	NS	NS	NS	NS
SILVER	MG/KG	1) 0.24	0.052	NS	NS	NS	NS
IRON	MG/KG	1) 9.5	44	NS	NS	NS	NS
POC							
ACETONE	MG/L	28000	T <500	32000	34000	35000	11000
BUTANOL	MG/L	1800	11000	<500	T <500	580	2500
ISO-BUTANOL	MG/L	1200	T <500	3100	3500	1800	T <500
DIOXANE	MG/L	2600	6400	1500	1400	1400	3100
ETHANOL	MG/L	27000	34000	15000	22000	31000	31000
ISOPROPANOL	MG/L	26000	56000	53000	27000	28000	40000
METHANOL	MG/L	35000	14000	45000	46000	56000	22000
METHYLENE CHLORIDE	MG/L	<500	<500	<500	<500	<T 500	<500
METHYL ETHYL KETONE	MG/L	590000	530000	330000	490000	740000	640000
TOLUENE	MG/L	10000	18000	25000	67000	12000	18000
P-XYLENE,M-XYLENE	MG/L	2200	23000	3500	12000	1700	8400
O-XYLENE	MG/L	T <500	4300	770	1900	<500	1500
TOTAL ORGANIC HALIDE	WT % Cl	17.21	1.11	36.87	24.42	17.25	0.25
EP TOX		NS	PASS	NS	NS	NS	NS
ARSENIC	MG/L	NS	<0.1	NS	NS	NS	NS
BARIUM	MG/L	NS	<1	NS	NS	NS	NS
CADMIUM	MG/L	NS	<0.2	NS	NS	NS	NS
CHROMIUM	MG/L	NS	<0.3	NS	NS	NS	NS
LEAD	MG/L	NS	<0.5	NS	NS	NS	NS
MERCURY	MG/L	NS	<0.1	NS	NS	NS	NS
SELENIUM	MG/L	NS	<1	NS	NS	NS	NS
SILVER	MG/L	NS	<0.2	NS	NS	NS	NS
TCLP		NS	#	NS	NS	NS	NS
FLASHPOINT	F	<75	NS	NS	NS	NS	NS
BOILING RANGE	C	62-90	NS	NS	NS	NS	NS
% CARBON	WT %	46.13	52.29	34.75	40.46	49.38	55.33
% HYDROGEN	WT %	9.14	11.48	6.59	7.76	9.79	11.21
% WATER	WT %	8.01	13.79	3.07	4.52	7.14	10.0
BTU CONTENT	BTU/LB	NS	11555	NS	NS	NS	NS

FOOTNOTE: 1) MG/L

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boiling range of this mixture was 60 to 81°C. A measurement of 3 weight percent total solids at 103°C, 100 percent volatile at 550°C, indicates that the mixture contains high boiling organic material but no inorganic ash. For this reason, metals and silica analyses were not performed.

The fact that the sum of the reported concentration of principal organic components, 1.54 g/ml, was greater than the measured density, 1.20 g/ml, may be attributed to the nonhomogeneity of the waste solvent. This material was dark brown-black in color and opaque with a very dirty appearance. It is likely that some of the individual sample aliquots taken to run different analytical tests were not representative of the bulk composition of the waste.

The filtered solvent influent of batch 2 (Table 7) was primarily isopropanol (690,000 mg/l) with small concentrations of ethanol and ortho-xylene reported. The mixture contained 3.66 percent water and less than 0.1 percent organic chlorine. Based on the measured density of 0.834 g/ml, the principal organic component (POC) analysis accounted for 83 percent of the sample mass. The boiling range of this waste was 73 to 93°C, which brackets isopropanol's normal boiling point of 82.5°C.

Residue analysis indicates only 0.16% total solids. However, since 2 percent of this amount, or 32 ppm, was nonvolatile, metals analyses were run. Less than 10 mg/kg of metals were found with iron, copper, and chromium present at detectable concentrations.

The filtered solvent influent of batch 3 (Table 8) was a mixture of three major components - methylene chloride (460,000 mg/l), methanol (300,000 mg/l), and methyl ethyl ketone (240,000) - with smaller but significant concentrations of acetone, butanol, dioxane, ethanol, isopropanol, toluene and xylene. The mixture also contained 3.59 percent water. Based on the measured density of 1.03 g/ml, principal organic components analysis accounted for 108 percent of the mass. This may be explained by sample non-homogeneity. Small rust-colored particles settled out from the thin amber colored liquid sample.

Residue analysis indicated about 70 mg/l inorganic solids, but less than 2 mg/l could be accounted for as iron and copper. No silica (an indicator of sand or dirt) was found. The boiling range of the waste solvent mixture was 44 to 85°C.

The predominant component of batch 4 (Table 9) filtered solvent influent was methyl ethyl ketone (MEK) which comprised 63 percent of the total mass. Other compounds detected were acetone, butanol, dioxane, ethanol, isopropanol, methanol, toluene, and xylenes. Principal organic component analysis left 23 percent of the mass unaccounted for, 8 percent of which was water. Given that chlorine analysis indicated 17.21% organic chlorine, it is obvious that one or more chlorinated organics were present but not identified by the POC analysis.

The waste contained less than 1 percent total solids at 103°C and had a relatively narrow boiling range of 62 to 78°C. MEK's normal boiling point is 79.6°C.

Product Characterization --

Analysis of the four product cuts taken during batch 1 showed that the lowest boiling component, freon, boiled off early as expected, appearing at highest concentration in the first cut (640,000 mg/l), decreasing concentrations in intermediate cuts, and absent from the final cut. High concentrations of 1,1,1-trichloroethane, and isopropanol were measured in the overhead product, although no trends were noticed for these chemicals among the different product cuts.

Analysis of product cuts for batch 2 show isopropanol concentrations increasing from 41% in the first cut to 86% of the final cut (percentages based on concentration of component divided by total density). Comparison of the carbon, hydrogen, chlorine, water, and density data indicates that one or more low-boiling, non-chlorinated and highly oxygenated compounds has been missed by the POC analysis. Methanol is a possible candidate.

Analysis of the four product cuts for batch 3 showed that the lowest boiling component - methylene chloride - boiled off early as expected, appearing at highest concentration in the first cut (1,100,000 mg/l) and decreasing concentration in subsequent cuts. A duplicate sample of the first product cut indicated a methylene chloride concentration of 910,000 mg/l.

Methanol concentration in the product was relatively constant. Methyl ethyl ketone - the least volatile of the three major components - was present at low concentration in the first two cuts and increased as distillation continued.

The organic chlorine analyses performed on batch 4 indicate that the unidentified chlorinated organic components boils over early, with its highest concentration in the first cut and decreasing concentrations in subsequent cuts. The level of methyl ethyl ketone is lowest in the initial cut (330,000 mg/l) and increases as distillation proceeds (640,000 mg/l).

The product cuts generated from the distillation process are rough cuts and as such are sold for their solvent value in noncritical products such as lacquer thinners, degreasers and glass cleaners.

Residue Characterization --

The still bottoms produced during batch 1, with a density of 0.95 g/ml, had carbon, hydrogen and organic chlorine compositions of 73.33%, 11.82% and 8.87% respectively. The gas chromatography analysis for principal organic components accounted for only 40 percent of the total sample mass, with most of this 40 percent identified as 1,1,1 trichloroethane. Dioxane, with a boiling point of 101°C - above the final still pot temperature for this batch, was concentrated in the still bottoms. The measurement of 71 percent volatile solids is an indicator of high boiling organic material, most likely oil and grease, due to the previous use of the solvent as a degreaser.

Despite its high BTU value (15,722 BTU/lb), low viscosity (4.5 centistokes) and low water content (0.04%), a chlorine content greater than five percent (8.85%) disqualified this residue from use as a fuel by Facility A's usual fuel blend customers. However, this material would have been accepted for fuel in a low-alkali cement kiln. The material failed the paint filter test, which is a test to identify the still bottom's suitability for direct landfill; therefore, it would require solidification prior to land disposal.

The still bottoms from the batch 2 waste isopropanol distillation, with a BTU value of 15,244 BTU/lb (100,189 BTU/gal), a water content of 2.86 percent and a chlorine content less than 0.1 percent, was acceptable for use in a fuel blend. This material had a density of 0.789 g/ml and a total volatile solids concentration of 7.1 percent, indicating high boiling organic components.

Principal organic components analysis accounted for only 46 percent of the total mass as isopropanol, xylene, and ethanol. The remaining organic material has a hydrogen to carbon ratio of 2.5 to 1.

Batch 3 still bottoms results are included in Table 8. With the exception of the lowest boiling component, methylene chloride, all compounds reported in the influent of batch 3 also appeared in the still bottoms from this batch. The high boilers - MEK, toluene and xylenes - were concentrated in the still bottoms. Water, at 7.13 percent, is also present at higher concentration in this residue than in the original waste. Principal organic component analysis accounted for 98 percent of the total mass.

Having a BTU value of 12,339 BTU/lb and chlorine content of 1.12 percent, this still bottoms was acceptable to be incorporated into Facility A's fuel blend. However, probably due to its large volume (94 gallons) and significant solvent value, this residue was saved for re-distillation as part of a future waste solvent batch.

The residue from distillation of the MEK waste (batch 4) was predominantly MEK with 13.79 percent water. The unidentified chlorinated organic which was present in the influent was depleted in the still bottoms, such that only 1.11 percent organic chlorine was measured in this residue. Of the 6.7 percent total solids, 2 percent or 1150 mg/l was nonvolatile inorganic material. 100 mg/kg silica, 44 mg/kg iron and 2.5 mg/kg copper were reported.

Facility A reported separating a wastewater layer from the still bottoms after gravity separation. The remaining organic residue would have a BTU value greater than the 11,555 BTU/lb measured in the mixed sample. This residue was incorporated into a fuel blend.

Evaluation of Effectiveness

In order to evaluate Facility A's batch distillation process, it is necessary to understand not only the engineering theory of the process discussed in this section, but also the facility's specific objectives.

Facility A's batch distillation process achieves the goals of recovering solvent and minimizing landfill of solvent wastes. Of the 1543 gallons of solvent-laden wastes processed during the week of June 24, 52 percent was recovered for sale as solvent, 4 percent was recovered for sale as fuel, 39 percent was retained for further processing, and only 3.4 percent was destined for landfill after solidification with vermiculite.

Batch distillation, as practiced at Facility A is applicable to the treatment of hazardous solvent wastes where the impurities rendering these solvents unsuitable for further direct use are high boiling materials (e.g., oils and greases) or nonvolatile solids (e.g., paint solids, grit, metals, polymeric resins).

If simple batch distillation is to be the sole treatment process, the recovered solvent must be acceptable for use, either recycled to the process which originally generated the waste solvent or in some other application. This effectively limits the types and concentrations of volatile impurities that can be accepted in the influent to a simple batch distillation process, but the limitation is end-use specific.

Facility A reduces this limitation and expands the applicability of their simple batch distillation process by recycling solvent recovered from the wastes of electronics industry generators to less critical end-uses for which virgin solvent purity is not required. For example, solvents processed during the week of June 24 included a batch of spent methyl ethyl ketone derived from a number of industrial sources (electronics, plating, specialty chemical manufacturing) and recovered for use in a paint thinner.

There are, however, some types of waste solvent impurities which present a problem for Facility A's distillation/recycling operation. Facility A's president and chemist reported two examples of solvent wastes which had been accepted for treatment at the facility in the past, but proved unsuitable for solvent recovery. One was a waste xylene containing paint resins and plasticizers. This material became rubbery in the still pot. A fluid layer was pumped off and blended into a secondary fuel mixture. The rubbery material was solidified with vermiculite for land disposal in drums.

The second problem waste was a spent toluene which contained mercaptans. The distilled product has a strong mercaptan odor, so it could not be sold as toluene. Therefore, it was blended into a secondary fuel mixture.

Currently, the major limitation on Facility A's batch distillation process is the maximum temperature achievable due to the limited capacity of the facility's steam boiler. Solvents with normal boiling points above 140°C cannot be distilled. However, Facility A plans to install a vacuum system, which will enable distillation of higher boiling solvents (e.g., perchloroethylene and kerosene) at reduced pressure.

Residue Management

The still bottoms from the four batch distillation runs each had sufficient fuel value to be incorporated in a fuel blend by the facility. However, the bottoms from the first batch were disqualified from use as a fuel by the facility because of its high chlorine content. The material passed the EP toxicity test but failed the paint filter test, therefore, it would require solidification prior to land disposal. The residue from distillation of the MEK waste in the fourth batch has a water content of 13.79 percent but the facility separated a wastewater layer by gravity separation thus the remaining organic residue would have a lower water content and greater BTU value than the mixed sample. This residue was incorporated in a fuel blend. The residue from batch three has sufficient heating value and a low enough chlorine content to be incorporated into a fuel blend but due to the large quantity of solvent remaining in the residue, the bottoms were saved to be re-distilled.

FACILITY D

Process Description

Facility D has been in operation since 1970, and currently offers solvent reclamation and distillation. Facility D accepts organic solvent wastes, which are generated by industries involved in metal finishing, solvent cleaning, lacquering and painting operations.

The distillation processes at Facility D are illustrated in Figure 3. Three stills are used to process organic liquids at Facility D: the main still, the batch kettle and the batch still reboiler. The operation of the stills can be changed in accordance with waste processing requirements. The stills are operated two shifts per day, 7:00 a.m. to 11:00 p.m. Two batches of organic liquids were processed through the main still at Facility D during the three day test program. One batch consisted of methyl ethyl ketone (MEK) mixed with paint pigment and the second batch was an oil-water emulsion. Two batches of mixed waste solvent were processed through the batch kettle and one batch of mixed waste solvent was processed in the batch still reboiler.

In general, incoming solvent wastes are pumped into storage tanks for clarification before distillation. Drum tops are removed after the liquids are pumped and settled solids are drained through a metal screen. The solids are sent to a landfill and the drained liquids are distilled. Paint cans are crushed, and the solids and liquids generated during crushing are mixed with absorbent materials for landfilling. Four 5000-gallon tanks, two 2000-gallon tanks, two 200-gallon tanks, and one 4000-gallon tank are located within a diked area with water nozzles for fire control. These tanks are used to store flammable liquids. One 4000-gallon tank is located outside the diked area and is used to store liquids with flash points above 200°F.

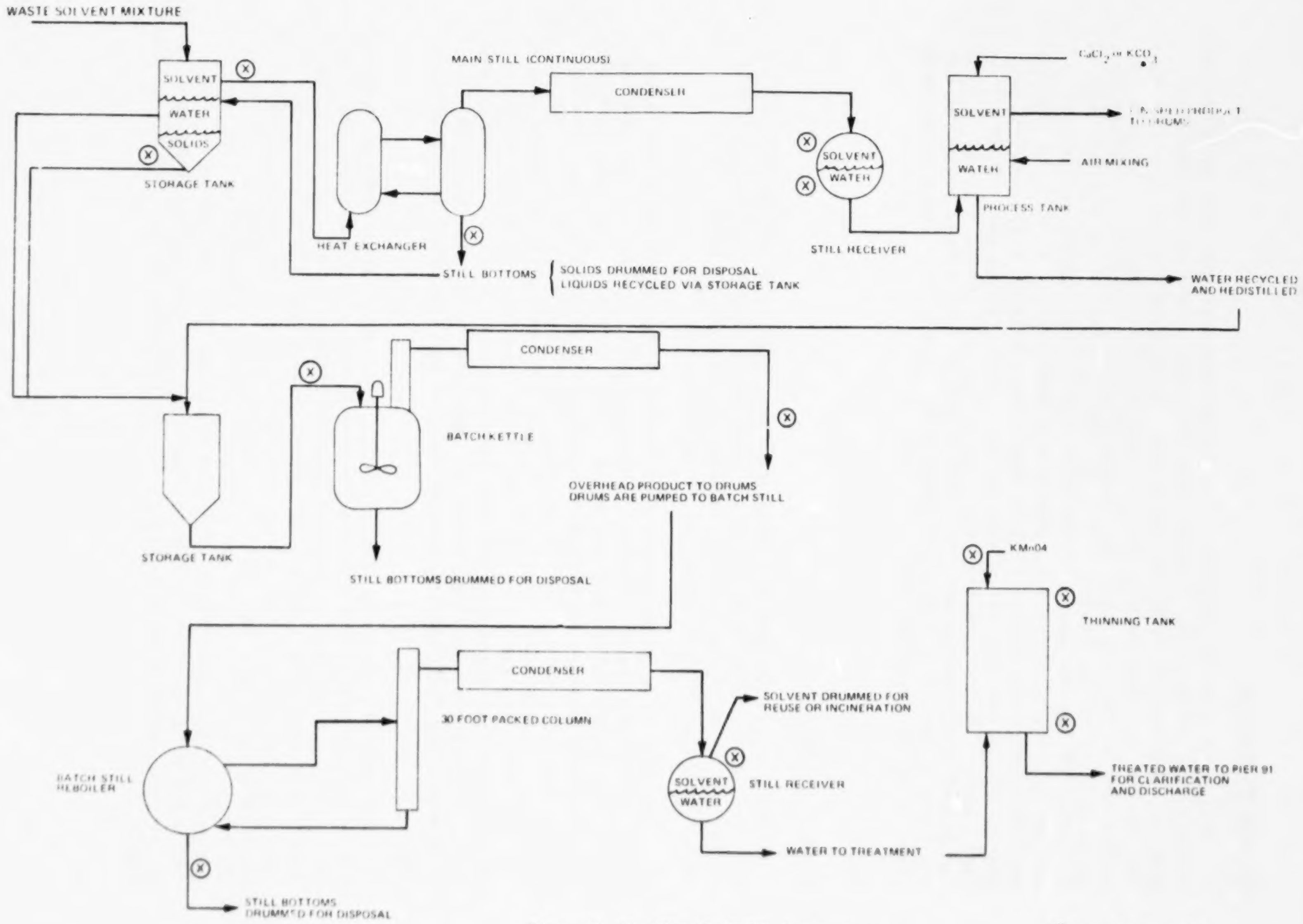
WASTE SOLVENT MIXTURE
SOLVENT
WATER
SOLIDS
STORAGE TANK
HEAT EXCHANGER
MAIN STILL (CONTINUOUS)
CONDENSER
SOLVENT
WATER
STILL RECEIVER
STILL BOTTOMS
SOLIDS DRUMMED FOR DISPOSAL
LIQUIDS RECYCLED VIA STORAGE TANK
$CaCl_2$ or KCO_3
SOLVENT
WATER
PROCESS TANK
[illegible] PRODUCT TO DRUMS
AIR MIXING
WATER RECYCLED AND REDISTILLED
CONDENSER
BATCH KETTLE
STORAGE TANK
STILL BOTTOMS DRUMMED FOR DISPOSAL
OVERHEAD PRODUCT TO DRUMS
DRUMS ARE PUMPED TO BATCH STILL
KMnO4
THINNING TANK
CONDENSER
30 FOOT PACKED COLUMN
SOLVENT DRUMMED FOR REUSE OR INCINERATION
SOLVENT
WATER
STILL RECEIVER
BATCH STILL REBOILER
WATER TO TREATMENT
TREATED WATER TO PIER 91 FOR CLARIFICATION AND DISCHARGE
STILL BOTTOMS DRUMMED FOR DISPOSAL
ⓧ SAMPLE LOCATIONS

Figure 3. Facility D distillation process

Solvents are pumped from the storage tank to the 500-gallon main still which is capable of continuous operation. The main still consists of two heat exchangers and one separator tank. The two shell and tube heat exchangers heat the wastes which flow into the separator tank. Typically one heat exchanger is operated in a semi-batch mode to treat 250 gallons of liquid while the second heat exchanger is cleaned. The main still is operated at 150°F under a vacuum of 15 to 20 inches of mercury. Vaporized liquid flows out of the separator, is cooled in a condenser and is collected in a 2000-gallon receiving tank. Liquid in the separator returns to the heat exchanger. Still bottoms may be recycled through the main still, processed further with the other stills or landfilled if the solids content is sufficient.

Water and solvent phases accumulate in the receiving tank and are transferred to one of two 5000-gallon process tanks. Calcium chloride or potassium carbonate can be mixed in the process tanks to dry the solvents. Recovered solvents from the process tanks are sold for reuse.

The water from the main still receiver and process tanks is transferred to another storage tank. The solid, water and mixed "rag" layers from the clarified solvent influent storage tank are combined with the process tank and receiver water. These mixed liquids are processed in a 1400-gallon batch distillation kettle, which is operated at 212°F. The overhead product containing water and solvents is collected in drums. The still bottoms are dumped approximately once each week and are sent to a landfill for disposal.

The overhead product from the kettle is distilled again in a 2000-gallon batch still equipped with a 30-foot refluxing column. The still is operated at 55°F under a vacuum of 15 to 20 inches of mercury. A falling water aspirator creates the vacuum for both the batch and main stills. Separate water and solvent phases are collected in a 200-gallon receiver. The mixed solvents may be sold for reuse or incinerated. Information on the extent of solvents being sold for reuse or being incinerated is not available. The water is transferred to one of two 2100-gallon "thinning" tanks and treated with potassium permanganate or alum. The treated water is transported to another TSD facility for clarification and discharge.

Still bottoms are distilled until they will solidify when cooled. Diatomaceous earth has been added in the past to solidify still bottoms, but this is not currently done. Cost of transporting the solidified material makes it currently more economical to solidify wastes at the landfill. Installation of a cement mixer at the facility is planned for future solidification requirements.

Facility D's goals for the distillation process were to recover solvents for reuse and/or to pretreat the waste so that it is acceptable for treatment by incineration, separate the water fraction and discharge accordingly, and produce a solid residue for landfill disposal. Operations of the three stills can be modified to accomplish these goals for varying waste compositions.

Cold and snowy weather during the test program caused freezing of the pipes therefore necessitating the still to shut down due to frozen pipes. As a result, no still bottoms were generated for the oil-water emulsion, and the still bottoms from the MEK waste were sampled before the process was complete.

Sampling Program

The on-site sampling program at Facility D took place during the week of November 18, 1985. The samples collected to evaluate the distillation of organic solvents represent three distillation operations, namely: the main still, the batch kettle and the batch still. The sampling locations are indicated in Figure 3.

Two batches of organic liquids - methyl ethyl ketone (MEK) mixed with paint pigment and an oil-water emulsion - were processed through the main still during the test program. Sample locations for the first organic liquid (MEK) included:

- clarified solvent influent;
- settled solvent solids from the receiving tank;
- finished water product from the still received;
- finished solvent product from the still receiver; and
- still bottoms from the separator.

Sample locations for distillation of the oil-water emulsion in the main still included:

- clarified solvent influent; and
- finished water product from the still receiver.

Two batches of mixed solvent waste were processed through the batch kettle during the test program. As Figure 3 shows, the influent to the batch kettle is typically a mixture of water and settled solids from the raw waste decanter tanks, and the water product from the main still. The sample locations for each batch run included:

- clarified solvent influent to the kettle; and
- four discrete finished product samples per raw waste sample from the receiver tank as a function of time.

The third distillation process monitored was the batch still. One batch of mixed solvent waste (MEK with paint pigment) was processed in the batch still reboiler during the test program. Samples taken to evaluate the batch still included:

- still bottoms from the batch still reboiler;
- finished solvent product collected in the still receiver; and
- a composited sample of the thinning tank contents.

No separate samples were taken of the batch still influent since the batch kettle finished solvent product was pumped directly to the batch still. Samples collected are summarized in Table 10.

Air monitoring of volatile organics was conducted in the following locations associated with the distillation process:

- near the batch kettle while sampling was taking place;
- above and below the batch kettle (closed unit);
- adjacent to an open top storage tank which contained wastewater, oil and solvents;
- at the condenser overflow pipe of the batch kettle operation; and
- near the heat exchanger form the main still.

Air quality samples were not collected because positive volatile organic readings (HNu meter) were not observed.

Data

Waste Characterization --

Two batches of organic liquids were processed through the main still. The first batch consisted of MEK mixed with paint pigment. The clarified solvent influent was a greyish-green single phase liquid containing a mixture of organic compounds with the predominant compound being MEK at 850,000 mg/l. Water accounted for 6.4% of the sample and 44,000 mg/l of organic compounds were unidentified by the gas chromatograph/mass spectrometer (GC/MS) scan conducted. Oil and grease represent a significant part of the sample by weight (180,000 ppm) as did the total solids (90,000 ppm). The nonvolatile solids (8,000 ppm) contained little silica, and low concentrations of metals; they indicate the presence of salts (2,000 ppm nonvolatile dissolved solids) and inorganic suspended solids (6,000 ppm nonvolatile suspended solids).

The second batch processed by the main still was a water and oil emulsion. The clarified solvent influent was a milky green, opaque liquid with a thin layer of brown settled solids on the bottom. The main organic component of the waste stream was 1,1,1-trichloroethane with a concentration of 210,000 mg/l. Ninety one percent of the sample was estimated as water with only two percent carbon. A significant portion of the carbon was accounted for by oil and grease which was present at a concentration of 15,000 ppm. The total solids concentration was 20,000 ppm and total volatile solids concentration was 19,000 ppm.

During the sampling episode, two batches of waste were treated by the batch kettle. The waste influent consisted of 1) water from the main still process tank and still receiver, and 2) water, solids and mixed "rag" layers from the clarified solvent influent storage tank.

The primary components measured in the influent stream were acetone at 84,000 mg/l, isopropanol at 67,000 mg/l and methyl ethyl ketone at 6,100 mg/l. The waste stream was 84 percent by weight water. The total solids

TABLE 10. SAMPLE LOCATIONS AND PROCEDURES - FACILITY D

Sample Reference	Description	Type of Sample	Time Interval
DISTILLATION			
Clarified Solvent Influent	Sampled from influent line going into main still or the batch kettle with a stainless steel bucket.	Grab	Once per batch
Settled Solvent Solids	Collected from a port in the storage tank bottom with a stainless steel bucket.	Grab	Once
Finished Solvent Product	Collected at a line feeding into drums from the overhead tank which follows each still.	Grab	Once per batch; one batch was sampled four times as a function of time.
Finished Water Product	Collected at a line feeding into drums from the overhead tank.	Grab	Once per batch
Still Bottoms	Collected from base of the heat exchanger with a stainless steel bucket.	Grab	Once per batch for 2 batches (main still and batch still reboiler)
Thinning Tank	One sample was collected by compositing samples from the bottom drain and the top of the tank.	Grab	Once for batch treated in batch still reboiler

concentration was only 5100 ppm, a fairly small quantity considering that the influent included settled solids from the clarified influent storage tank. The total volatile solids content was 4,400 ppm. Metals found at low concentrations included copper (1.8 mg/l), iron (0.4 mg/l) and zinc (1.4 mg/l).

The influent stream to the batch still consisted of the overhead product from the two batch kettle runs. No discrete influent sample was taken of this waste stream since the product streams of the two batch kettle runs were sampled. The composition of this stream is therefore covered in the product characterization of the batch kettle process.

Product Characterization --

The main still aqueous product contained highly soluble organics such as acetone, ethanol, isopropanol and methanol. Methanol, with a concentration of 9,900 mg/l, was the most concentrated of the alcohols and ketones detected in the aqueous product. Water was 60 percent of the sample by weight, and only 1,600 mg/l of unidentified organic compounds were estimated from the GC/MS chromatograph.

The concentration of MEK in the finished solvent product was enriched to 910,000 mg/l. The water content was measured at thirteen percent while carbon and hydrogen accounted for 63 and 11 percent of the sample by weight, respectively.

For the water and oil emulsion batch which was treated by the main still only 1 sample was taken of the overhead product (Table 11). The aqueous product included two phases: a cloudy yellow liquid composing 98 percent of the volume and a pale yellow phase floating on top with some solids present. The sample contained only 0.2 percent water by weight. The major constituent measured in the product was 1,1,1-trichloroethane at 910,000 mg/l. The levels of MEK, methyl isobutyl ketone (MIBK) and toluene were higher in the finished product than in the clarified solvent influent. The total organic halide concentration measured 21.8 percent, up from 1.2 percent in the clarified solvent influent.

Table 12 presents the finished solvent product results for the two batches of waste treated by the batch kettle. The finished solvent product from batch one of the batch kettle run contained high concentrations of acetone, isopropanol and MEK, which were the major constituents in the influent. Isopropanol and acetone were concentrated in the first cut of the finished product at concentrations of 110,000 mg/l and 92,000 mg/l respectively. The concentrations of these organics diminish with each succeeding sample. As the temperature of the still pot increased with time, the percent of water increased from 83 percent in the first sample to 96 percent in the final sample.

The finished solvent product for batch two contained primarily methyl ethyl ketone at a concentration of 700,000 mg/l. Isopropanol (100,000 mg/l), ethanol (64,000 mg/l) and acetone (69,000 mg/l) were also present in high concentrations. Water accounts for 44 percent of the sample. The second and third product cuts contain appreciable concentrations of organics and

TABLE 11. MAIN STILL ANALYTICAL RESULTS - FACILITY D

POLLUTANT	UNITS	BATCH 1 - MEK SOLVENT CLARIFIED SOLVENT INFLUENT (10600)	FINISHED WATER PRODUCT (10604)	FINISHED SOLVENT PRODUCT (10605)	STILL BOTTOMS (10602)	BATCH 2 OIL-WATER EMULSION CLARIFIED SOLVENT INFLUENT (10660)	FINISHED PRODUCT (10664)
PRINCIPAL ORGANIC COMPOUNDS							
acetone	mg/l	17,000	8,500	20,000	T <2,500	6,400	4,700
n-butanol	mg/l	6,500	<500	6,000	11,000	510	T <2,500
dioxane	mg/l	<2,500	<500	<2,500	<2,500	40,000	4,100
ethanol	mg/l	<2,500	8,400	<2,500	<2,500	T <500	<2,500
ethyl acetate	mg/l	10,000	<500	13,000	4,100	<500	<2,500
isopropanol	mg/l	27,000	9,000	41,000	9,600	3,500	T <2,500
methanol	mg/l	<2,500	9,900	<2,500	<2,500	18,000	<2,500
methyl ethyl ketone (MEK)	mg/l	850,000	4,400	910,000	500,000	9,100	72,000
methyl isobutyl ketone (MIBK)	mg/l	21,000	<500	20,000	30,000	T <500	2,100
1,1,1-trichloroethane	mg/l	<2,500	<500	<2,500	<2,500	210,000	**910,000
toluene	mg/l	48,000	<500	51,000	41,000	1,200	4,100
total unidentified concentration	mg/l	44,000	1,600	25,000	99,000	3,300	<2,500
METALS							
cadmium	mg/l	8	NS	NS	* 620	<0.2	NS
total chromium	mg/l	22	NS	NS	* 2,200	0.6	NS
copper	mg/l	4	NS	NS	* 270	5.0	NS
iron	mg/l	16	NS	NS	* 1,200	75.0	NS
lead	mg/l	16	NS	NS	* 2,200	1.0	NS
nickel	mg/l	<0.3	NS	NS	* <130	<0.3	NS
zinc	mg/l	10	NS	NS	* 490	3.0	NS
OTHER ANALYSIS							
chemical oxygen demand	mg/l	NS	NS	NS	I	NS	NS
total organic halide	weight % Cl	0.4024	0.002	0.470	0.500	1.220	21.830
oil & grease	ppm	180,000	NS	NS	260,000	15,000	NS
total solids	ppm	90,000	NS	NS	310,000	20,000	NS
total volatile solids	ppm	82,000	NS	NS	290,000	19,000	NS
total suspended solids	ppm	26,000	NS	NS	200,000	5,200	NS
total dissolved solids	ppm	64,000	NS	NS	110,000	15,000	NS
total volatile suspended solids	ppm	20,000	NS	NS	170,000	4,300	NS
total volatile dissolved solids	ppm	62,000	NS	NS	120,000	15,000	NS
silica	weight %	0.03	NS	NS	0.07	0.17	NS
% water	weight %	6.43	59.94	12.82	3.48	90.69	0.19
flashpoint	DEG. F	<75	NS	NS	NS	<75	NS
boiling range	DEG. C	75 - 95	NS	NS	NS	99 - 100	NS
% Carbon	weight %	65	4	63	68	2	23
% Hydrogen	weight %	11	7	11	10	11	3
Btu content	Btu/lb	NS	NS	NS	14,188	NS	NS
viscosity	cst	NS	NS	NS	4.3 @52C	7.7 @1C	NS
paint filter test	see note	NS	NS	NS	FAIL	NS	NS

* = units are mg/kg
I = sample interference
NS = not sampled
T = trace concentration detected below the average reporting limit
note: result based on qualitative observation

** per phone conversation 7/15/86 with analytical lab

TABLE 12. BATCH KETTLE DISTILLATION RESULTS - FACILITY D

		BATCH #1					BATCH #2				
POLLUTANT	UNITS	CLARIFIED SOLVENT INFLUENT (10610)	FINISHED SOLVENT PRODUCT (10614)	FINISHED SOLVENT PRODUCT (10615)	FINISHED SOLVENT PRODUCT (10616)	FINISHED SOLVENT PRODUCT (10617)	CLARIFIED SOLVENT INFLUENT (10630)	FINISHED SOLVENT PRODUCT (10634)	FINISHED SOLVENT PRODUCT (10635)	FINISHED SOLVENT PRODUCT (10636)	FINISHED SOLVENT PRODUCT (10637)
PRINCIPAL ORGANIC COMPOUNDS											
acetone	mg/l	84,000	92,000	20,000	4,100	3,200	80,000	69,000	36,000	16,000	4,000
n-butanol	mg/l	<2,500	<2,500	T <2,500	<2,500	<2,500	<2,500	10,000	5,800	4,900	2,600
ethanol	mg/l	<2,500	<2,500	<2,500	<2,500	<2,500	<2,500	64,000	93,000	78,000	43,000
isobutanol	mg/l	<2,500	<2,500	<2,500	<2,500	<2,500	<2,500	2,600	T <2,500	<2,500	<2,500
isopropanol	mg/l	67,000	110,000	35,000	12,000	10,000	60,000	100,000	110,000	75,000	27,000
methanol	mg/l	<2,500	<2,500	<2,500	<2,500	<2,500	<2,500	<2,500	19,000	22,000	17,000
methyl ethyl ketone (MEK)	mg/l	6,100	4,900	T <2,500	T <2,500	T <2,500	4,900	700,000	220,000	97,000	26,000
methyl isobutyl ketone (MIBK)	mg/l	T <2,500	T <2,500	<2,500	<2,500	<2,500	<2,500	24,000	3,000	T <2,500	T <2,500
1,1,1-trichloroethane	mg/l	<2,500	<2,500	<2,500	<2,500	<2,500	<2,500	14,000	<2,500	<2,500	<2,500
trichloroethylene	mg/l	<2,500	<2,500	<2,500	<2,500	<2,500	<2,500	T <2,500	<2,500	<2,500	<2,500
toluene	mg/l	<2,500	<2,500	<2,500	<2,500	<2,500	<2,500	44,000	T <2,500	<2,500	<2,500
total unidentified concentration	mg/l	5,700	4,800	2,700	<2,500	<2,500	3,600	16,000	<2,500	3,000	2,800
METALS											
cadmium	mg/l	<0.2	NS	NS	NS	NS	<0.2	NS	NS	NS	NS
total chromium	mg/l	<0.3	NS	NS	NS	NS	0.5	NS	NS	NS	NS
copper	mg/l	1.8	NS	NS	NS	NS	3.0	NS	NS	NS	NS
iron	mg/l	0.4	NS	NS	NS	NS	2.0	NS	NS	NS	NS
lead	mg/l	<0.5	NS	NS	NS	NS	1.5	NS	NS	NS	NS
nickel	mg/l	<0.3	NS	NS	NS	NS	<0.3	NS	NS	NS	NS
zinc	mg/l	1.4	NS	NS	NS	NS	2.0	NS	NS	NS	NS
OTHER ANALYSIS											
total organic halide	weight % Cl	0.033	0.140	0.003	0.027	0.020	0.030	1.420	0.380	0.160	0.300
oil & grease	mg/l	5,000	NS	NS	NS	NS	2,700	NS	NS	NS	NS
total solids	mg/l	5,100	NS	NS	NS	NS	5,000	NS	NS	NS	NS
total volatile solids	mg/l	4,400	NS	NS	NS	NS	4,100	NS	NS	NS	NS
total suspended solids	mg/l	70	NS	NS	NS	NS	340	NS	NS	NS	NS
total dissolved solids	mg/l	5,000	NS	NS	NS	NS	4,700	NS	NS	NS	NS
total volatile suspended solids	mg/l	66	NS	NS	NS	NS	260	NS	NS	NS	NS
total volatile dissolved solids	mg/l	4,300	NS	NS	NS	NS	3,800	NS	NS	NS	NS
silica	weight %	0.06	NS	NS	NS	NS	0.21	NS	NS	NS	NS
% water	weight %	83.60	83.36	89.44	95.75	95.99	82.42	43.81	47.40	77.34	89.13
flashpoint	DEG. F	<75	NS	NS	NS	NS	<75	NS	NS	NS	NS
boiling range	DEG. C	74 - 106	NS	NS	NS	NS	80 - 110	NS	NS	NS	NS
% Carbon	weight %	9	21	8	8	11	9	27	28	26	9
% Hydrogen	weight %	11	12	11	12	11	11	11	11	12	11
viscosity	cst	1.1 @3.5C	NS	NS	NS	NS	1.1 @2.5C	NS	NS	NS	NS

NS = NOT SAMPLED
T = TRACE CONCENTRATIONS DETECTED BELOW THE AVERAGE REPORTING LIMIT

the water content increases substantially from 47 percent in the second cut to 77 percent in the third cut. By the fourth product cut, the organic concentrations had decreased with ethanol (43,000 mg/l) being the predominant organic component. Water accounted for 89 percent by weight of the sample.

The finished solvent product for the batch still reboiler (Table 13) was 89 percent by weight water. The predominant organics present in the product were isopropanol (47,000 mg/l) and ethanol (29,000 mg/l).

The aqueous overhead product from the batch still is sent to a thinning tank where potassium permanganate is added to oxidize organic compounds and alum is added to flocculate suspended solids before the treated product is transported for further treatment at another Facility D location. The results indicate that levels of methylene chloride and acetone were 250 mg/l and 180 mg/l respectively. There is a fairly high oil and grease concentration of 40,000 ppm and total volatile solids of 19,000 ppm. Iron (11 mg/l) was the only metal detected at concentrations above the detection limits.

Residue Characterization --

Results for the still bottoms generated by the distillation processes were included in Tables 11-13 which were presented earlier. In the still bottoms from the MEK batch treated by the main still, higher boiling organic compounds such as methyl isobutyl ketone (MIBK) and n-butanol were more concentrated with respect to the more volatile compounds like acetone. MEK remained the principal organic compound detected, accounting for 500,000 mg/l. Roughly 99,000 mg/l of unidentified organic compounds were accounted by peaks on the sample chromatograph. The BTU content of the still bottoms was 14,200 BTU/lb. Water only constituted three percent of the still bottoms by weight. Non-volatile oil and grease accounted for 260,000 ppm. The chlorine content of the still bottom was 0.5%. The total solids concentration is high (310,000 ppm), and almost 94 percent of these solids are volatile. All seven metals analyzed, except nickel, were detected and concentrations were one to two orders of magnitude higher than the clarified solvent influent concentrations. Lead and chromium were found in the greatest concentration, each at 2,200 mg/kg.

There were no samples taken of the still bottoms from the main still for batch two (oil and water emulsion) and still bottoms produced from the operation of the batch kettle were not available to be sampled.

The still bottoms generated from the batch still reboiler consisted of ninety-five percent water, two percent carbon and a small quantity of oil and grease (420 ppm). Total solids were measured at 7,400 ppm and total volatile solids were 1,400 ppm. The chlorine content of the batch still reboiler bottoms was 0.03%. Dioxane was found in the greatest concentration (13,000 mg/l). All other principal organic compounds were below 10,000 mg/l. Metal analysis detected 27 mg/l of copper and small quantities of cadmium (0.3 mg/l), iron (1.0 mg/l) and zinc (4.0 mg/l).

TABLE 13. BATCH STILL REBOILER RESULTS-FACILITY D

POLLUTANT	UNITS	STILL BOTTOMS (10642)	FINISHED SOLVENT PRODUCT (10645)	THINNING TANK (10710)
VOLATILE ORGANIC COMPOUNDS				
methylene chloride	ug/l	NS	NS	250,000
acetone	ug/l	NS	NS	180,000
2-butanone (MEK)	ug/l	NS	NS	31,000
1,1,1-trichloroethane	ug/l	NS	NS	20,000
4-methyl-2-pentanone	ug/l	NS	NS	3,600
toluene	ug/l	NS	NS	1,100
PRINCIPAL ORGANIC COMPOUNDS				
acetone	mg/l	2,600	7,200	NS
n-butanol	mg/l	1,700	4,500	NS
dioxane	mg/l	13,000	17,000	NS
ethanol	mg/l	10,000	29,000	NS
isobutanol	mg/l	T <500	<2,500	NS
isopropanol	mg/l	7,400	47,000	NS
methanol	mg/l	5,800	<2,500	NS
methyl ethyl ketone (MEK)	mg/l	4,900	11,000	NS
methyl isobutyl ketone (MIBK)	mg/l	T <500	<2,500	NS
1,1,1-trichloroethane	mg/l	2,000	<2,500	NS
total unidentified concentration	ppm	23,000	4,400	NS
ACID EXTRACTABLE COMPOUNDS				
2-nitrophenol	ug/l	NS	NS	24,000
BASE/NEUTRAL EXTRACTABLE CMPDS				
isophorone	ug/l	NS	NS	5,800
METALS				
cadmium	mg/l	0.3	NS	<0.6
total chromium	mg/l	<0.3	NS	<0.9
copper	mg/l	27.0	NS	<1.8
iron	mg/l	1.0	NS	11.0
lead	mg/l	<0.5	NS	<1.5
nickel	mg/l	<0.3	NS	<0.9
zinc	mg/l	4.0	NS	<0.9
OTHER ANALYSIS				
chemical oxygen demand	mg/l	I	NS	NS
total organic carbon	mg/l	NS	NS	12,000
total organic halide	weight % Cl	0.030	0.065	0.000
oil & grease	mg/l	420	NS	1,600
total solids	mg/l	7,400	NS	40,000
total volatile solids	mg/l	1,400	NS	19,000
total suspended solids	mg/l	130	NS	1,300
total dissolved solids	mg/l	7,300	NS	39,000
total volatile suspended solids	mg/l	110	NS	820
total volatile dissolved solids	mg/l	1,300	NS	18,000
silica	weight %	0.13	NS	0.56
% water	weight %	95.33	88.65	NS
% Carbon	weight %	2	6	NS
% Hydrogen	weight %	11	11	NS
Btu content	Btu/lb	<200	NS	NS
viscosity	cst	2.2 @67C	NS	NS
paint filter test	see note	FAIL	NS	NS

I = SAMPLE INTERFERENCE
NS = NOT SAMPLED

FOR VOLATILES, ACID AND BASE/NEUTRAL EXTRACTABLES, POLLUTANTS IN TABLE 5 AND NOT LISTED ABOVE WERE NOT DETECTED IN ANY OF THE SAMPLES ABOVE THE ANALYTICAL DETECTION LIMIT

NOTE: RESULT BASED ON QUALITATIVE OBSERVATION

Evaluation of Effectiveness

In order to evaluate the solvent recovery system both the solvent treatment system as a whole, and the individual processes that comprise the system have been evaluated. It should be noted however, that these evaluations are based on limited data. Due to unusual and severe winter weather, plant operations were curtailed and samples of some process residuals were not obtained.

The residuals from the process include the still bottoms from the main still, batch kettle and batch still reboiler; liquid fractions include a dried finished solvent product, treated water, and a mixed solvent stream. Samples of the dried finished product and the batch kettle bottoms were not available.

Overall Treatment System --

The process reclaimed solvent for reuse or incineration; produced water for discharge to the sewer system, and reduced the quantity of material for landfill disposal. The final aqueous stream contained high concentrations of methylene chloride (250 mg/l) and acetone (180 mg/l) after treatment (Table 13).

Main Still --

The influent to the main still is the top solvent layer from the storage tank. The main still separates high boiling materials (e.g. oils and greases) and nonvolatile solids (e.g. paint solids, grit, metals, polymeric resins) from the lower boiling materials. The separation is crude because all vapors generated from the liquid are condensed in one equilibrium stage and no purification of product is attempted.

The analytical results in Table 11 show that the concentration of oil and grease in the MEK waste increased from 180,000 ppm in the influent to 260,000 ppm in the bottoms, and the concentration of total solids increased from 90,000 ppm to 310,000 ppm. Since the bottoms sample was obtained "prematurely" these numbers are most likely lower than would be expected for the final solid bottoms. This partially explains the quantities of recoverable organics, especially MEK remaining in the bottoms. The weight percent of water in the finished product is relative high at 13 percent by weight compared to six percent in the influent and three percent in the bottoms. This increase results from boiling water out of relatively heavy non-volatiles in the bottoms. The bottoms have a fairly high BTU content (14,188 BTU/lb), low chlorine content (0.05% Cl), and low weight percent of water which potentially may be suitable for use as a fuel blend.

The presence of heavy metals, such as lead (2,200 mg/kg) could, however, preclude its use as a fuel. The still bottom solids are ultimately drummed for disposal, and the liquid fraction of the bottoms waste is recycled to a storage tank for further distillation by the main still.

Some concentration of organics in the finished solvent product has occurred. However, the solvent product produced is still a mixture of several organic compounds. It would be acceptable for reuse in the original process or in another application that does not require virgin purity. Both the reclaimed solvent and overhead water require further treatment. The solvent must be dried to reduce the water content, and the water must be treated to oxidize residual organic compounds.

Solvent Process Tank --

In order to remove some of the water from the solvent, both the reclaimed solvent and the overhead water are pumped into a 5000 gallon process drying tank. Calcium chloride or potassium carbonate is added to the tank during air agitation. Water is removed from the solvent and this salting out process results in a drier solvent product. Samples of the dried solvent and water phases were not obtained due to process interruptions caused by the weather.

Batch Kettle --

The influent to the batch kettle is the water, solids and mixed rag layers from the storage tank as well as the water from the main still. The batch kettle separates impurities including high boiling compounds and nonvolatiles from organics and water. The low boiling organics are further separated from water in the batch still and the batch still receiver where separate organic and water phases form. Analytical results from four different samples of the overhead product obtained over time indicate that the initial overhead samples are substantially more concentrated in organics than the influent. Subsequent samples show gradual diminishing organic content with an increasing percentage of water present. No sample of the still bottoms was obtained; however, it is presumed that the oil and grease, metals and solids would have been concentrated in this material.

Batch Still Reboiler --

Overhead products from the batch kettle are temporarily stored in 55 gallon drums, then pumped to the batch still reboiler. A composite sample from the batch still was not obtained because samples of the kettle's overhead product has already been collected. The batch still is fitted with a 30 ft. packed tower to provide a number of equilibrium stages and achieve separation of organics from water. The bottoms from this still were 95 percent water (Table 13) and had a very low BTU content (less than 200 BTU/lb) and low chlorine content (0.03 percent Cl). The low heating value would preclude its use as fuel, and the high organic concentration would require additional treatment. The solvent layer collected in the receiver still contained 89 percent water by weight. It is assumed that the high water content would not enhance reuse or incineration as an ultimate disposal method.

Thinning Tank --

The overhead water from the batch still is pumped to a thinning tank for oxidation of organics by potassium permanganate. The effluent from this tank is trucked to another Facility D treatment facility for clarification, oxidation of residual organics, and ultimately discharge to the sewer system. The treated water contained high concentrations of methylene chloride (250 mg/l), acetone (180 mg/l), MEK (31 mg/l), 1,1,1-trichloroethane (20 mg/l), 2-nitrophenol (24 mg/l) and isophorone (5.6 mg/l). Treatment of these organic compounds in the aqueous waste with potassium permanganate did not appear to be complete.

Residue Management

The solid still bottoms generated from the main still distillation process are drummed for disposal and sent to a landfill while the drained liquids are recycled to the storage tanks to be treated again by the main still. The still bottoms generated by the batch kettle and batch still reboiler are also drummed for disposal and sent to be landfilled. Generally the still bottoms produced from the distillation processes at Facility D during the sampling episode had a low chlorine content. The main still produced a bottoms with a higher BTU content and higher suspended solids content than that of the batch still reboiler. The bottoms from both processes failed the paint filter test which serves as an indication that the still bottoms are not suitable for direct landfill. Facility D reported that still bottoms are heated until solid and only solid still bottoms are drummed for landfill disposal. No solid still bottoms were generated during the sampling episode thus this operating procedure cannot be confirmed by this data.

SECTION 6

STEAM DISTILLATION

THEORY

Steam distillation is a process which uses direct injection of steam to enable distillation of organic liquids at temperatures below their normal boiling points. The process can effect very high percentage recoveries of water-immiscible volatile solvents from wastes containing nonvolatile impurities. The technique is especially useful when the material to be distilled is thermally unstable, contains nonvolatile contaminants which would foul heat transfer surfaces in an ordinary batch still or has a very high boiling point.

The principle of operation governing steam distillation is Dalton's Law of Partial Pressures, which states that each component in a mixture of immiscible liquids exerts its own vapor pressure independent of the other components.

$$\bar{p}a = \frac{Y_a R T n}{V}$$

$\bar{p}a$ = partial pressure of component A in mixture

ya = mole fraction of component A in mixture

R = Universal Gas Constant

T = Temperature of mixture

n = number of moles in mixture

V = Total Volume of mixture

At any given system pressure, the mixture boils at a temperature lower than the boiling point of any component (thus, always less than 100°C or 212°F for steam distillation). The vapor phase mole fraction of any component is equal to the ratio of its vapor pressure to the total system pressure, regardless of its liquid phase mole fraction.

Steam distillation is generally carried out as a semibatch operation. The feed is first charged to a still pot and then steam is continuously charged through spargers in the bottom of the still. The liquid in the still pot is heated by condensation of steam until the sum of the organic and water vapor pressures is equal to the system pressure and the mixture begins to boil. The mixed vapors rise and are condensed in a water-cooled heat exchanger. The distillate is collected in an accumulator tank. Gravity settling in this tank results in the separation of two phases - aqueous and organic. If water is soluble in the organic liquid, a subsequent drying operation may be necessary to purify the solvent product. The aqueous phase contains dissolved organics and must be treated or discharged as a wastewater stream.

As steam distillation proceeds, the concentration of organic solvent in the still pot decreases until all that remains is a mixture of nonvolatiles and water (condensed steam). At this point the temperature of the boiling liquid in the pot will increase to the boiling point of water and the process should be terminated. The residual still bottoms, then, are typically a mixture of water and nonvolatiles (e.g. - solids, oil, and grease) containing only a very small concentration of the volatile solvent.

Both the temperature at which steam distillation of a given solvent will occur and the composition of the distillate can be estimated from a simple vapor pressure vs. temperature plot called a Hausbrand diagram. A Hausbrand diagram for 1,1,1-trichloroethane is presented in Figure 4. The diagram plots the difference between total system pressure and water vapor pressure vs. temperature. Solvent vapor pressure vs. temperature curves are plotted on the same graph.

The intersection of the water curve with the vapor pressure curve for a solvent indicates the temperature at which steam distillation can take place. The ratio of solvent vapor pressure to water vapor pressure at this temperature is equal to the molar ratio of solvent to water in the distillate.

Thus, according to the Hausbrand diagram, a waste solvent containing only 1,1,1-TCA contaminated with high boiling oil and grease would be steam distilled at 65°C, 9 degrees below the normal boiling point. The distillate would contain 2.0 moles 1,1,1-TCA per mole of water, which is equivalent to 21.5 lb 1,1,1-TCA per lb of water or 15.8 gallons 1,1,1-TCA per gallon of water.

FACILITY C

Process Description

Facility C is a commercial solvent recycling plant which has two steam distillation systems, one dedicated to nonchlorinated solvents (still no. 1, 120-gallon batch capacity), and the other to chlorinated solvents (still no. 2, 80-gallon batch capacity). Each is a "Dyna 1" system, manufactured by DCI Corporation and consisting of a horizontal stainless-steel still pot set up for direct steam injection, a water-cooled condenser and a condensate coalescer or accumulator tank. Figures 5 and 6 are diagrams depicting the nonchlorinated and chlorinated steam distillation processes, respectively. The operation of both steam distillation systems is described below.

Nonchlorinated waste solvent is usually received by tank truck at Facility C. This solvent is pumped from the tank truck to a 12,000 gallon conical bottom cylindrical tank. On occasion drummed nonchlorinated solvent

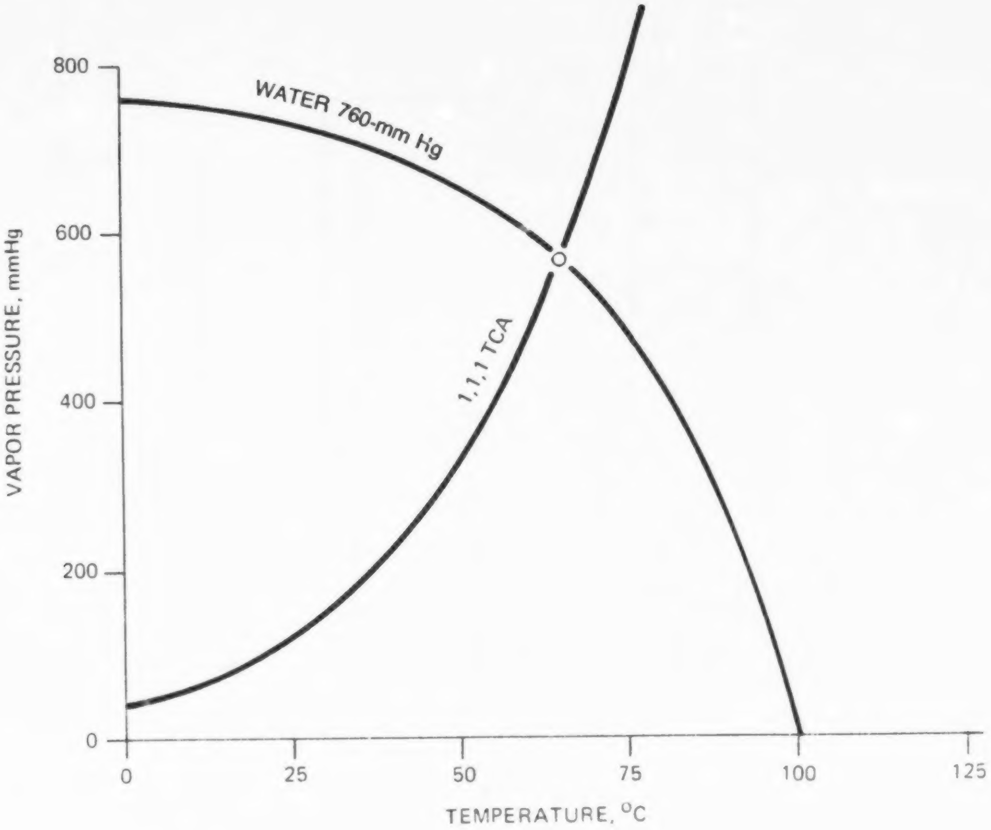

Figure 4. Hausbrand diagram for 1,1,1-trichloroethane at 1 atm (760 mmHg).

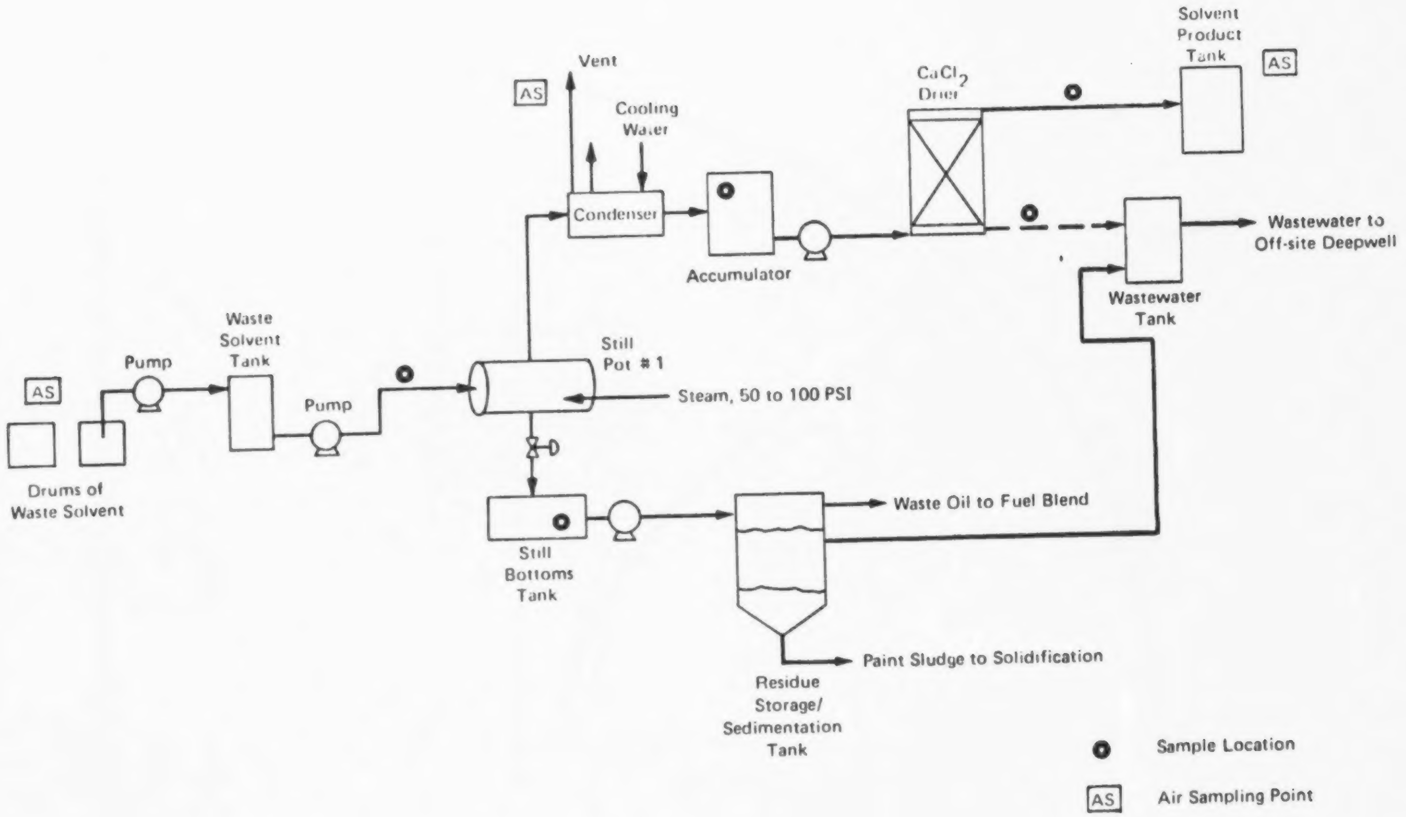
Drums of Waste Solvent
AS
Pump
Waste Solvent Tank
Pump
Still Pot # 1
Steam, 50 to 100 PSI
Still Bottoms Tank
Vent
AS
Cooling Water
Condenser
Accumulator
CaCl2 Drier
Solvent Product Tank
AS
Wastewater Tank
Wastewater to Off-site Deepwell
Waste Oil to Fuel Blend
Paint Sludge to Solidification
Residue Storage/ Sedimentation Tank
Sample Location
AS
Air Sampling Point

Figure 5. Flow diagram/sample points for steam distillation at Facility C, nonchlorinated slovents.

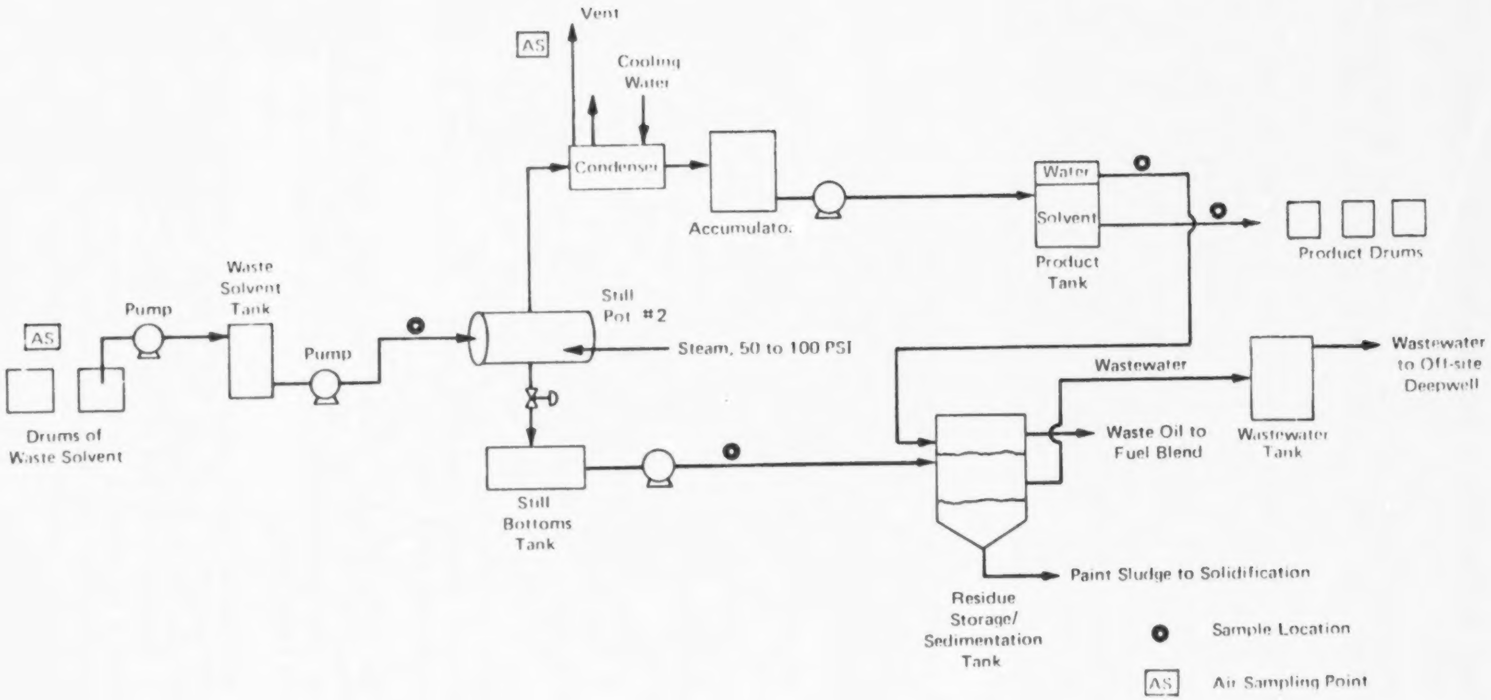
Vent
AS
Cooling Water
Condenser
Accumulator
Water
Solvent
Product Tank
Product Drums
AS
Pump
Waste Solvent Tank
Pump
Still Pot #2
Steam, 50 to 100 PSI
Drums of Waste Solvent
Still Bottoms Tank
Wastewater
Wastewater to Off-site Deepwell
Waste Oil to Fuel Blend
Wastewater Tank
Paint Sludge to Solidification
Residue Storage/ Sedimentation Tank
Sample Location
AS
Air Sampling Point

Figure 6. Flow diagram /sample points for steam distillation at Facility C, chlorinated solvents

is received at Facility C and this material is pumped into the above mentioned tank.

Chlorinated waste solvent is received in 55-gallon drums. Drums of waste solvent are pumped with a drum pump into a 1000-gallon horizontal feed tank.

The "Dyna 1" system operates in an automatic semibatch manner. At the beginning of a steam distillation cycle, the system is in "feed mode". During "feed mode", a valve in the waste solvent feed line is opened automatically and waste solvent is pumped into the horizontal still pot from the appropriate waste solvent tank. After a preset time the unit switches to "recirculation mode", causing the feed valve to close and the recirculation valve to open. In addition, the steam valve opens, allowing the waste solvent to be directly injected with 50-60 psi steam from Facility C's oil-fired boiler. Thus, the waste solvent is recirculated continuously to provide mixing for good heat transfer and suspension of solids while the batch is distilled.

The solvent/water vapors that evaporate from the still pot are condensed in the water-cooled condenser and collect in the coalescer or accumulator tank. A liquid level pump on/off control system periodically discharges about 6 gallons of condensed product.

In the case of nonchlorinated solvents, the liquid exiting the accumulator enters near the bottom of a calcium chloride dryer and flows upward through a packed bed of calcium chloride. The calcium chloride dissolves, causing a phase separation to occur between the solvent and the calcium chloride-saturated water. The denser aqueous phase collects at the bottom of the dryer. The less dense solvent layer floats on top of the aqueous layer and exits the dryer near the top. The aqueous layer is allowed to collect in the bottom of the dryer until a sufficient level has accumulated. The aqueous layer is then blown down and out the bottom of the dryer and sent to a 8800-gallon cylindrical wastewater holding tank. This wastewater is eventually trucked away for disposal in an off-site deepwell. The solvent product exiting the top of the dryer is sent to a 6800-gallon cylindrical solvent product holding tank. This solvent product is returned to the generator by tank truck.

In the case of chlorinated solvents, liquid exiting the accumulator flows through coils in the solvent feed tank - recovering waste heat and preheating the feed - before being discharged to the 1000-gallon horizontal product storage tank.

Two distinct layers separate in the product storage tank, a denser chlorinated solvent layer on the bottom and an aqueous phase floating on top. When the product storage tank has reached a sufficient level, the solvent is drained out the bottom of the tank through a hole into 55-gallon drums. When the solvent layer has been completely drained, as indicated by a color change when the aqueous layer appears, drum filling is terminated. The aqueous layer remaining in the product tank is then pumped to the waste residue holding tank.

For both the nonchlorinated and chlorinated systems, the end of a batch steam distillation cycle occurs when the still pot contents reach a preset temperature (195°F for nonchlorinated, 205°F for chlorinated). This temperature is measured by a thermocouple in the still pot and recorded on the temperature vs. time chart on the control panel. At this point, the steam valve closes and the drain valve on the bottom of the still pot opens. The still bottoms drain into a small tank below the still pot and are pumped to the 11,000-gallon conical bottom cylindrical waste residue tank. The same residue tank receives still bottoms from both stills.

The drain valve closes automatically after a set amount of time. The system then returns to the feed mode and continues sequential batch operation until manually stopped. Non-chlorinated solvent batch cycles generally required between 50 to 60 minutes during the operations observed. Chlorinated solvent batch cycles required 30 to 40 minutes. For both types of solvents, another 10 minutes was required to drain the still bottoms, refill the still pot, and heat the still contents before distillation commenced.

Material in the waste residue tank is allowed to phase separate into three distinct layers - sludge, water, and oil. When this tank is nearly full, the sludge layer is first pumped out of the bottom of the tank into a roll-off container to be solidified with fly ash on-site. Next, the aqueous layer is pumped into the wastewater tank for off-site disposal by deepwell injection. The oily layer is then pumped to a 7500-gallon round-bottom cylindrical tank. This material is blended with other waste oils and eventually sold as a fuel blend.

The principal goals of the stema distillation process at Facility C were:

1. To recover the maximum quantity of recyclable solvent from spent solvents and still bottoms. Facility C does not attempt to fractionate solvents to high purity specifications; rather, they make a rough cut to separate clean, clear solvent from nonvolatile components like oils, grease and paint solids. The solvent products are usually returned to the waste generator for reuse in noncritical applications like degreasing and paint line clean-out.

2. To produce a free-flowing still bottom which can be pumped from the steam stripper still to the residue tank.

Sampling Program

The on-site sampling program at Facility C took place during a three day period in October 1985. Steam distillation sample points are indicated on Figures 5 and 6. They include waste solvent influent, solvent product, still bottoms and condensate wastewater. Since the solvent product sample will be taken downstream of the $CaCl_2$ drier a sample of the ketone influent to the drier will be taken to evaluate the effectiveness of the $CaCl_2$ drier. Table 14 provides descriptions of these sample points and the procedures used to procure samples.

TABLE 14. FACILITY C SAMPLE LOCATIONS AND PROCEDURES

Sample Reference	Description	Type of Sample	Time Interval
Non-Chlorinated Waste Solvent Influent	Samples were taken from the sample tap located on the feed line to the still while the feed pump was in "feed" mode. A beaker was used to take the sample aliquots to make up the composite.	Composite	Four aliquots of 500 ml were taken at about 1.5-2 hr. intervals as the still was in "feed" mode. Aliquots were composited in a glass jar to form a single sample per day.
Solvent Product	Samples were taken from the in-line product tap located after the $CaCl_2$ drier. A beaker was used to take the sample aliquots to make up the composite.	Composite	Four aliquots of 500 ml were taken at 1.5 hour intervals and were composited to form a single sample per day.
Ketone Influent to Dryer	Samples were collected from the sample port on the small accumulator box. A beaker was used to take the sample aliquots to make up the composite.	Composite	Four aliquots of 25 ml were taken at 1.5 hour intervals and were composited in a glass jar to form a single sample per day.

TABLE 14. (Continued) FACILITY C SAMPLE LOCATIONS AND PROCEDURES

Sample Reference	Description	Type of Sample	Time Interval
Still Bottoms	Samples were collected thru a sample hatch on top of the bottoms drain tank. The drain tank was located beneath the still pot. A COLIWASA was used to collect the aliquot samples.	Composite	Four aliquots of 750 ml were taken at about 1.5-2 hr. intervals at the time when the still bottoms were drained. These aliquots were composited in a glass jar to form a single sample per day.
Condensate Wastewater	Samples were collected from the wastewater sample tap on the $CaCl_2$ dryer after the wastewater had reached a sufficient level in the dryer. A 5-gal plastic carboy container was used to collect the sample.	Grab	2 gallon sample was collected after the wastewater had reached a sufficient level in the dryer.
Chlorinated Waste Solvent Influent	Samples were taken from the sample tap located on the feed line to the still while the feed pump was in "feed" mode. A beaker was used to take the sample aliquots to make up the composite.	Composite	Four aliquots of 50 ml were taken at about 1.5-2 hour intervals as the still was in "feed" mode. Aliquots were composited in a glass jar to form a single sample per day.

TABLE 14. (Continued) FACILITY C SAMPLE LOCATIONS AND PROCEDURES

Sample Reference	Description	Type of Sample	Time Interval
Solvent Product	Product solvent was intermittently (5 min) pumped from the accumulator to the product solvent holding tank. Phase separation of the solvent and wastewater in the horizontal holding tank was achieved. After about 5 hours of operation an operator drained the bottom solvent layer into drums. A sample was collected from this drain line.	Grab	One 2000 ml sample was collected from a hose draining the product solvent tank after the still had been in operation for about five hours.
Still Bottoms Storage Tank	Sample was taken at a pressure relief valve located on top of tank while nonchlorinated still bottoms were being discharged to this tank. A glass sample bulb was used.	Grab	One sample, Day Two.
Condensate Wastewater	After the solvent layer had all been drained from the holding tank, as determined by color change in the liquid, the less dense wastewater was drained. A sample was collected from this drain line.	Grab	One 2 gallon sample was collected from a hose draining the product solvent/wastewater holding tank after the still had been in operation for about 5 hours.

TABLE 14. (Continued) FACILITY C SAMPLE LOCATIONS AND PROCEDURES

Sample Reference	Description	Type of Sample	Time Interval
Still Bottoms	Samples were taken from in-line tap on the pipe leading from the still to the residue holding tank. A beaker was used to take the sample aliquots to make up the composite.	Composite	Four 750 ml aliquot samples were collected at about 1.5-2 hour intervals. Aliquots were composited in a glass jar to form a single sample per day.

Data

Waste Characterization --

Facility C accepts and processes both chlorinated and nonchlorinated solvent wastes for treatment by steam distillation. During the sampling program two different types of solvent waste were treated. These were:

(1) a spent methyl ethyl ketone solvent blend generated by a paint line clean-out process at an automobile manufacturing facility, and

(2) still bottoms from the fractional distillation of chlorinated degreasing solvent.

Table 15 summarizes generator's manifest descriptions and qualitative observations for these wastes. Note that the waste nonchlorinated solvent treated and sampled during the first two days of sampling was the same material.

Analytical data for the wastes and for all other steam distillation samples is summarized in Tables 16 through 18. The major solvent component of the nonchlorinated waste solvent influent was methyl ethyl ketone, which made up 53 percent of the total sample mass in the day 1 sample and 61 weight percent of the sample collected on day 2. Other solvents identified in the waste on one or both days were isopropanol, methyl isobutyl ketone, toluene, 1,1,1-trichloroethane, and p-xylene. The density of the waste averaged 0.857 g/ml and its water content averaged about 5 percent by weight. Metals analysis showed measurable quantities of copper, iron, lead, zinc and titanium. Titanium, present at 195 mg/l in the day 1 sample and 11 mg/l in the day 2 sample, is present as the white pigment titanium dioxide in almost all paints. Therefore its presence in this spent paint line clean-out solvent was not surprising.

The major solvent component of the day 3 chlorinated waste was 1,1,1 trichloroethane which made up 32 percent of the sample weight. Dioxane was the only other organic compound reported at a measurable concentration.

The total organic chlorine measurement of 54 percent is not in agreement with the principal organic component analysis, suggesting either that the measured value for 1,1,1-trichloro-ethane (32 percent) may be in error or that another chlorinated organic compound was present in the waste but not identified by the POC analysis.

The total solids concentration was 180,000 mg/l and BTU content was 8,267 BTU/lb. Six metals were reported at greater than 1 mg/l. Iron at 160 mg/l and zinc at 44 mg/l were the major metals.

Product Characterization --

The nonchlorinated solvent product was a clear, free-flowing liquid with a yellow tinge. The analytical data indicates that the day 1 product

TABLE 15. ORGANIC SOLVENTS TREATED DURING SAMPLING PROGRAM

Sampling Date	Generator Industry Type	Generator's Manifest Description	RCRA Waste Code	Comments and Observations
10/1/85 (Day 1)	Auto Parts Manufacturing	Waste Solvent, Flammable Liquid	D001	Two phase liquid. Top layer, about two-thirds the total volume, is black liquid. Bottom layer is clear, yellowish liquid. Free-flowing, strong ketone odor.
10/2/85 (Day 2)	Auto Parts Manufacturing	Waste Solvent, Flammable Liquid	D001	Two phase liquid. Top layer, about two-thirds the total volume, is black liquid. Bottom layer is clear, yellowish liquid. Free-flowing, strong ketone odor.
10/3/85 (Day 3)	Solvent Reclaimer	Waste 1,1,1-Trichloroethane	F002	Dark black-brown opaque liquid One phase. Fairly thin, free-flowing.

Table 16. ANALYTICAL DATA - STEAM DISTILLATION - NON-CHLORINATED DAY 1

PARAMETER	UNITS	WASTE SOLVENT INFLUENT 10433	KETONE INFLUENT TO DRYER 10434	SOLVENT PRODUCT 10435	CONDENSATE WASTEWATER 10436	STILL BOTTOMS 10437
Viscosity	cSt	NM	NM	NM	NM	2.6
Paint Filter Test		NM	NM	NM	NM	FAIL
Specific Gravity		.867	NM	.810	.815	*6).991
COD	mg/L	NS	NS	NS	1) 120000	two phase
TOC	mg/L	NS	NS	NS	32000	NS
Total Solids	mg/L	59000	NS	1000	450000	8200
Total Dissolved Solids	mg/L	58000	NS	990	450000	7400
Total Suspended Solids	mg/L	1100	NS	26	960	850
Total Volatile Solids	mg/L	59000	NS	940	150000	6700
Total Vol Sus Solids	mg/L	1000	NS	12	350	820
Total Vol Dis Solids	mg/L	58000	NS	910	150000	5900
Silica	wt %	0.14	NS	NS	NS	0.17
METALS						
Cadmium	mg/L	<0.2	NS	NS	NS	<0.2
Chromium	mg/L	0.8	NS	NS	NS	<0.8
Copper	mg/L	2	NS	NS	NS	1.6
Iron	mg/L	41	NS	NS	NS	25
Lead	mg/L	1.6	NS	NS	NS	1.2
Nickel	mg/L	1.2	NS	NS	NS	2
Silver	mg/L	<0.2	NS	NS	NS	<0.2
Tin	mg/L	<1	NS	NS	NS	<1
Titanium	mg/L	195	NS	NS	NS	<1
Zinc	mg/L	4	NS	NS	NS	10
VOC						
acetone	ug/L	NS	NS	NS	1200000	NS
2-butanone	ug/L	NS	NS	NS	<50000	NS
EXTRACTABLE ORGANICS						
ACID COMPOUND	ug/L	NS	NS	NS	<10	NS
BASE/NEUTRALS	ug/L	NS	NS	NS	<10	NS
PESTICIDES	ug/L	NS	NS	NS	<0.1	NS
POC						
acetone	mg/L	T <5000	NS	T <5000	NS	<5000
n-butanol	mg/L	T <5000	NS	T <5000	NS	1200
isobutanol	mg/L	T <5000	NS	T <5000	NS	570
isopropanol	mg/L	9300	NS	30000	NS	790
methanol	mg/L	<5000	NS	<5000	NS	780
methyl ethyl ketone	mg/L	460000	NS	700000	NS	5200
methyl isobutyl ketone	mg/L	6800	NS	15000	NS	<5000
toluene	mg/L	42000	NS	58000	NS	<5000

Table 16. ANALYTICAL DATA - STEAM DISTILLATION - NON-CHLORINATED DAY 1

PARAMETER	UNITS	WASTE SOLVENT INFLUENT 10433	KETONE INFLUENT TO DRYER 10434	SOLVENT PRODUCT 10435	CONDENSATE WASTEWATER 10436	STILL BOTTOMS 10437
1,1,1-trichloroethane	mg/L	50000	NS	110000	NS	<5000
EP TOX		NS	NS	NS	NS	PASS

Arsenic	mg/L	NS	NS	NS	NS	<0.1
Barium	mg/L	NS	NS	NS	NS	1
Cadmium	mg/L	NS	NS	NS	NS	0.4
Chromium	mg/L	NS	NS	NS	NS	<0.3
Lead	mg/L	NS	NS	NS	NS	<0.5
Mercury	mg/L	NS	NS	NS	NS	<0.1
Selenium	mg/L	NS	NS	NS	NS	<1
Silver	mg/L	NS	NS	NS	NS	<0.2
Total Chlorine	%	0.77	NS	0.76	15.47	<0.1
Total Inorganic Chloride	%	58 ppm	NS	44 ppm	17.98	202
Total Organic Chlorine(TOX)	%	0.77	NS	0.76	0.6	<0.1
Flashpoint	F	<75	NS	NS	NS	NS
Boiling Range	C	70-130	NS	54-92	NS	NS
% carbon	%	64.55	NS	68.05	NS	6.91
% hydrogen	%	10.38	NS	11.08	NS	11.04
% water	%	5.81	4.97	0.79	NS	91.82
BTU content	btu/lb	13441	NS	NS	NS	WNB

FOOTNOTES
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*6 MULTIPHASE

SYMBOLS

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# = A DECISION WAS MADE NOT TO PERFORM THE ANALYSIS DESPITE THE ORIGINAL INTENTION AS OUTLINED IN THE TEST PLAN
WNB = WOULD NOT BURN
FOR PRINCIPLE ORGANIC COMPOUNDS, VOLATILES, BASE-NEUTRALS AND ACID EXTACTABLES, POLLUTANTS NOT LISTED WERE NOT DETECTED IN ANY OF THE SAMPLES ABOVE THE ANALYTICAL DETECTION LIMIT

Table 17. ANALYTICAL DATA - STEAM DISTILLATION - NON-CHLORINATED DAY 2

PARAMETER	UNITS	WASTE SOLVENT INFLUENT 10442	KETONE INFLUENT TO DRYER 10443	SOLVENT PRODUCT 10444	CONDENSATE WASTEWATER 10445	CONDENSATE WASTEWATER 10457	STILL BOTTOMS 10446
Viscosity	cSt	NM	NM	NM	NM	NM	NM
Paint Filter Test		NM	NM	NM	NM	NM	FAIL
Specific Gravity		.847	NM	.826	1.24	1.23	*6) .946
COD	mg/L	NS	NS	NS	130000	90000	340000
TOC	mg/L	NS	NS	NS	23000	22000	NS
Total Solids	mg/L	30000	NS	2800	470000	470000	260000
Total Dissolved Solids	mg/L	27000	NS	2800	470000	470000	260000
Total Suspended Solids	mg/L	3100	NS	8.4	280	2300	210
Total Volatile Solids	mg/L	27000	NS	340	160000	150000	250000
Total Vol Sus Solids	mg/L	2800	NS	5.2	90	740	210
Total Vol Dis Solids	mg/L	24000	NS	330	160000	150000	250000
Silica	mg/L	0.16	NS	NS	NS	NS	*
METALS -------							
Cadmium	mg/L	<0.2	NS	NS	NS	NS	<1
Chromium	mg/L	0.8	NS	NS	NS	NS	<3
Copper	mg/L	1.6	NS	NS	NS	NS	3
Iron	mg/L	25	NS	NS	NS	NS	92
Lead	mg/L	1.2	NS	NS	NS	NS	<26
Nickel	mg/L	1.2	NS	NS	NS	NS	11
Silver	mg/L	<0.2	NS	NS	NS	NS	<2
Tin	mg/L	*	NS	NS	NS	NS	<81
Titanium	mg/L	11	NS	NS	NS	NS	<1500
Zinc	mg/L	5	NS	NS	NS	NS	<16
VOC ----							
acetone	ug/L	NS	NS	NS	590000	690000	NS
2-butanone	ug/L	NS	NS	NS	42000000	44000000	NS
EXTRACTABLES --------------							
ACID COMPOUNDS	ug/L	NS	NS	NS	<600	<60	NS
BASE/NEUTRAL							
isophorone	ug/L	NS	NS	NS	<600	T <60	NS
PESTICIDES	ug/L	NS	NS	NS	<0.1	<0.1	NS
POC -------							
acetone	mg/L	T <5000	NS	T <5000	NS	NS	49
n-butanol	mg/L	T <5000	NS	T <5000	NS	NS	1300
dioxane	mg/L	<5000	NS	<5000	NS	NS	65
ethanol	mg/L	<5000	NS	<5000	NS	NS	160
isobutanol	mg/L	T <5000	NS	T <5000	NS	NS	650
isopropanol	mg/L	T <12000	NS	6100	NS	NS	820

Table 17. ANALYTICAL DATA - STEAM DISTILLATION - NON-CHLORINATED DAY 2

PARAMETER	UNITS	WASTE SOLVENT INFLUENT 10442	KETONE INFLUENT TO DRYER 10443	SOLVENT PRODUCT 10444	CONDENSATE WASTEWATER 10445	CONDENSATE WASTEWATER 10457	STILL BOTTOMS 10446
methanol	mg/L	640	NS	<5000	NS	NS	23
methyl ethyl ketone	mg/L	520000	NS	780000	NS	NS	35000
methyl isobutyl ketone	mg/L	9900	NS	8100	NS	NS	350
toluene	mg/L	70000	NS	43000	NS	NS	110
1,1,1-trichloroethane	mg/L	<5000	NS	<5000	NS	NS	<5
p-xylene	mg/L	130000	NS	<5000	NS	NS	100
m-xylene	mg/L	<5000	NS	<5000	NS	NS	280
EP TOX -------		NS	NS	NS	NS	NS	PASS
Arsenic	mg/L	NS	NS	NS	NS	NS	<0.1
Barium	mg/L	NS	NS	NS	NS	NS	<1
Cadmium	mg/L	NS	NS	NS	NS	NS	<0.5
Chromium	mg/L	NS	NS	NS	NS	NS	<0.3
Lead	mg/L	NS	NS	NS	NS	NS	<0.5
Mercury	mg/L	NS	NS	NS	NS	NS	<0.1
Selenium	mg/L	NS	NS	NS	NS	NS	<1
Silver	mg/L	NS	NS	NS	NS	NS	<0.2
Total Chlorine	%	2.75	NS	<0.4	15.80	16.00	3.85
Total Inorganic Chloride	%	91	NS	<28 ppm	19.83	20.10	0.024
Total Organic Chlorine(TOX)	%	2.75	NS	<0.4	<0.6	<0.6	3.83
Flashpoint	F	LT 75	NS	NS	NS	NS	NS
Boiling Range	C	58-92	NS	70-90	NS	NS	NS
% carbon	%	66.72	NS	67.39	NS	NS	4.35
% hydrogen	%	11.03	NS	11.06	NS	NS	11.05
% water	%	4.07	6.82	4.23	NS	NS	90.54
BTU content	btu/lb	13659	NS	NS	NS	NS	WNB

SYMBOLS

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FOR PRINCIPLE ORGANIC COMPOUNDS,VOLATILES,BASE-NEUTRALS AND ACID-EXTRACTABLES, POLLUTANTS NOT LISTED WERE NOT DETECTED IN ANY OF THE SAMPLES ABOVE THE ANALYTICAL DETECTION LIMIT

Table 18. ANALYTICAL DATA - STEAM DISTILLATION - CHLORINATED DAY 3

PARAMETER	UNITS	WASTE SOLVENT INFLUENT 10447	SOLVENT PRODUCT 10448	CONDENSATE WASTEWATER 10449	STILL BOTTOMS 10450	STILL BOTTOMS 10458
Viscosity	cSt	NM	NM	NM	11.7	11.7
Paint Filter Test		NM	NM	NM	FAIL	FAIL
Specific Gravity		1.18	1.32	1.02	0.939	.935
TOC	mg/L	NS	NS	26000	NS	NS
COD	mg/L	NS	NS	1) 85000	IF	IF 1.4
Total Solids	mg/L	180000	750	950	500000	540000
Total Dissolved Solids	mg/L	170000	740	920	440000	500000
Total Suspended Solids	mg/L	5400	6.0	31	57000	45000
Total Volatile Solids	mg/L	170000	750	510	500000	540000
Total Vol Sus Solids	mg/L	4600	4.0	5	52000	42000
Total Vol Dis Solids	mg/L	170000	750	510	440000	500000
Silica	wt %	0.042	NS	NS	0.022	0.12
METALS						
Cadmium	mg/L	0.6	NS	NS	23	21
Chromium	mg/L	0.7	NS	NS	29	27
Copper	mg/L	9	NS	NS	22	18
Iron	mg/L	160	NS	NS	552	320
Nickel	mg/L	1.3	NS	NS	2	1.5
Lead	mg/L	3	NS	NS	3	2
Silver	mg/L	0.2	NS	NS	<0.2	<0.2
Titanium	mg/L	10	NS	NS	23	17
Zinc	mg/L	44	NS	NS	108	84
VOC						
methylene chloride	ug/L	NS	NS	100000	NS	NS
1,1,1-trichloroethane	ug/L	NS	NS	440000	NS	NS
trichloroethene	ug/L	NS	NS	270000	NS	NS
EXTRACTABLE ORGANICS						
ACID COMPOUNDS	ug/L					
2,4-dimethylphenol	ug/L	NS	NS	T <800	NS	NS
phenol	ug/L	NS	NS	9100	NS	NS
BASE/NEUTRALS	ug/L	NS	NS	<800	NS	NS
PESTICIDES	ug/L	NS	NS	<0.2	NS	NS
POC						
dioxane	mg/L	32000	39000	NS	<10000	<10000
toluene	mg/L	T <5000	T <5000	NS	<10000	<10000
1,1,1-trichloroethane	mg/L	380000	1400000	NS	<10000	<10000

Table 18. ANALYTICAL DATA - STEAM DISTILLATION - CHLORINATED DAY 3

PARAMETER	UNITS	WASTE SOLVENT INFLUENT 10447	SOLVENT PRODUCT 10448	CONDENSATE WASTEWATER 10449	STILL BOTTOMS 10450	STILL BOTTOMS 10458
trichloroethene	mg/L	<5000	940000	NS	<10000	<10000
EP TOX		PASS	NS	NS	FAIL	FAIL

Arsenic	mg/L	<0.1	NS	NS	<0.1	<0.1
Barium	mg/L	<1	NS	NS	<1	<1
Cadmium	mg/L	<0.5	NS	NS	1.6	1.5
Chromium	mg/L	<0.3	NS	NS	7	7
Lead	mg/L	<0.5	NS	NS	20	20
Mercury	mg/L	<0.1	NS	NS	<0.1	<0.1
Selenium	mg/L	<1	NS	NS	<1	<1
Silver	mg/L	<0.2	NS	NS	<0.2	<0.2
Total Chlorine	%	53.99	71.25	0.24	6.82	6.77
Total Inorganic Chloride	%	0.011	80 ppm	<0.1	815 ppm	907 ppm
Total Organic Chlorine(TOX)	%	53.98	71.25	0.24	6.74	6.68
Flashpoint	F	<75	NS	NS	NS	NS
Boiling Range	C	48-65	37-92	NS	NS	NS
% carbon	%	36.09	24.61	NS	53.06	53.31
% hydrogen	%	4.85	2.89	NS	10.74	11.16
% water	%	0.94	1.54	NS	25.72	26.01
BTU content	btu/lb	8267	NS	NS	11364	11581

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FOR PRINCIPLE ORGANIC COMPOUNDS, VOLATILES, BASE-NEUTRALS AND ACID-EXTRACTABLES, POLLUTANTS NOT LISTED WERE NOT DETECTED IN ANY OF THS SAMPLES ABOVE THE ANALYTICAL DETECTION LIMIT consisted of 86 percent methyl ethyl ketone, while the day 2 product was 94.4 percent methyl ethyl ketone. Water content ranged from 0.79 weight percent on day 1 to 4.23 weight percent on day 2. The average product density was 0.818 g/ml.

The chlorinated solvent product from day 3 was primarily 1,1,1-trichloroethane with a measured density of 1.32. Principal organic component analysis actually reported the 1,1,1-trichloro-ethane concentration as 106 percent of the sample mass, whereas the total organic chlorine measurement of 71.25 weight percent is consistent with 89.3 percent 1,1,1-TCA.

The total solids concentration of the product was less than 1 percent that of the waste influent.

Residue Characterization --

Steam distillation results in two residues - the aqueous phase of the condensed overhead vapor - "condensate wastewater" - and the still bottoms. Condensate wastewater resulting from steam distillation of the nonchlorinated solvent waste on days 1 and 2 had an average of 460,000 mg/l dissolved solids most of which can be attributed to dissolved calcium chloride from the drying operation. The inorganic chloride measurement was approximately 19 percent by weight. The total organic carbon concentration in this wastewater was measured at 32,000 mg/l in the day 1 sample and 23,000 mg/l on day 2. Acetone was detected in both samples - at 1200 mg/l on day 1 and 600 mg/l on day 2. Methyl ethyl ketone, the major component of the solvent waste, was reported in the day 2 sample at a concentration of 43,000 mg/l. This value is well below the aqueous solubility of methyl ethyl ketone which is 353,000 mg/l at 77°F. The high calcium chloride concentration may reduce the solubility of solvents in the wastewater. Surprisingly, methyl ethyl ketone was not detected in the day 1 condensate wastewater.

Still bottoms from the nonchlorinated waste treatment contained about 90 weight percent water. Due to the high water content this material would not burn and therefore would not be acceptable as a fuel substitute.

The condensate wastewater from steam distillation of chlorinated solvent waste contained high concentrations of three chlorinated solvents: 100 mg/l methylene chloride, 440 mg/l 1,1,1-trichloroethane, and 270 mg/l trichloroethene. Phenol, an acid extractable priority pollutant, was measured at 9,100)g/l.

The still bottoms residue from steam distillation of the chlorinated solvent waste contains about 500,000 mg/l total solids and has a density, 0.935 mg/l, less than that of water.

The water content of this residue is about 26 weight percent. Some of the metals in the waste solvent are concentrated in the still bottoms. Cadmium and chromium, reported at concentrations less than 1 mg/l in the feed, appear in the bottoms at concentrations more than thirty times higher. Copper, iron, titanium and zinc concentrations in the still bottoms are more than double the waste solvent influent concentrations, while nickel, lead and silver are reported at approximately the same low concentrations in both the influent and the bottoms.

The still bottoms failed the EP Toxicity test with cadmium, chromium and lead which were all present at concentrations higher than acceptable under the EP Toxicity Test Procedure (46 CFR 35247, July 7, 1981). The fuel value of this material was about 11,500 BTU/lb.

Evaluation of Effectiveness

In order to evaluate Facility C's steam distillation process and to compare it to the other solvent treatment operations, it is necessary to understand not only the engineering theory of the process discussed above, but also the facility's specific objectives and circumstances.

The principal goals of the steam distillation process at Facility C were:

1. To recover the maximum quantity of recyclable solvent from spent solvents and still bottoms. Facility C does not attempt to fractionate solvents to high purity specifications; rather, they make a rough cut to separate clean, clear solvent from nonvolatile components like oils, grease and paint solids. The solvent products are usually returned to the waste generator for reuse in noncritical applications like degreasing and paint line clean-out.

2. To produce a free-flowing still bottom which can be pumped from the steam stripper still to the residue tank.

The steam distillation process results in three residue streams - wastewater, waste oil, and paint sludge. The availability of an on-site solidification process and landfill is a special circumstance which enables Facility C to handle the paint sludge residue easily and inexpensively. Waste oil is incorporated into fuel blends and sold. The wastewater residue does present a significant problem, since it typically contains percentage levels of organic solvent compounds and cannot be treated in Facility C's on-site aqueous waste treatment process. This wastewater is periodically trucked off-site for disposal by deepwell injection.

Steam distillation, as practiced at Facility C, is applicable to the treatment of hazardous solvent wastes where the impurities rendering these solvents unsuitable for further use are high boiling materials (e.g., oils and greases) or nonvolatile solids (e.g., paint solids, grit, metals, polymeric resins).

Facility C's process can recover usable solvent from solvent wastes where the impurities are nonvolatiles such as oil, grease, and paint solids but the wastewater generated still contains a significant level of organics which must be disposed of. Data from the sampling program indicates that the steam distillation process showed good recovery for both chlorinated (1,1,1-trichloroethane) and non-chlorinated (methyl ethyl ketone) solvents. In both cases the residual bottoms contained a significant percentage of water and was free-flowing; no problems were encountered in pumping still bottoms to the residue tank and no clean-out of the still pot was required between batches.

As compared to other non-fractionating solvent recovery technologies - i.e., simple distillation and thin film evaporation - steam distillation achieves greater percentage recoveries of solvent from a given waste, uses less energy, and may require less maintenance. However, the generation of solvent-laden wastewater residues is a notable disadvantage unique to direct-contact steam distillation. In order to select the best process for a given application, the costs and environmental impact of disposing of this residue would have to be weighed against the value of additional recovered solvent the energy cost savings, and the maintenance cost savings. Cost information was not obtained during the sampling program.

Residue Management

As already described, steam distillation results in two residues - the condensate wastewater and the still bottoms. Still bottoms tend to have very high concentrations of water. At Facility C, still bottoms from both stills are pumped to an 11,000-gallon conical bottom cylindrical waste residue tank. The condensate wastewater from chlorinated solvent processing is also pumped to this tank.

Gravity separation in this tank results in three distinct layers - sludge, water, and oil. The sludge is solidified with fly-ash and landfilled on-site. The water layer is combined with nonchlorinated process condensate wastewater and shipped off-site for disposal in a deepwell. The oil layer is blended with other waste oils to be sold as a fuel substitute.

SECTION 7

AGITATED THIN FILM EVAPORATION AND SCREW AUGER EVAPORATION

Agitated thin film evaporation (ATFE) is a process capable of concentrating nonvolatile components of waste mixtures. The technique is especially useful in cases where fouling, foaming, high viscosity, thermal degradation or other factors present potential operating problems to other technologies. This is a direct result of the high mass transfer rates achieved through turbulence. The ATFE has the ability to attain high heat transfer rates with viscous liquids. The disadvantages of the ATFE are the high amount of maintenance required and the small treatment capacity of the ATFE which both can result in high costs. Three agitated thin film evaporation processes, one at Facility J, one at Facility K, and one at Facility L, were included in the sampling program. A summary of the types of wastes treated at these three facilities is presented in Table 19.

The heated screw auger is an analogous technology designed to strip residual solvent from viscous still bottoms, drum heels or any other high solids content waste. This process was observed at Facility K.

THEORY

Agitated Thin Film Evaporator

Liquid waste is fed to the agitated thin film evaporator (ATFE) where longitudinal blades mounted on a motor driven rotor maintain the waste against the heat transfer surface; i.e., the inside wall of the cylindrical vessel. This surface is enclosed in a heating jacket which usually employs steam or hot oil as the heating medium (temperatures up to 650 deg F).

The agitation and liquid film are maintained by the blades as they move along the heat transfer surface. The blade tips typically travel at 30 to 40 feet per second at clearances of 0.007 to 0.10 inch which creates high turbulence. This facilitates efficient heat and mass transfer, shortens required waste residence time, and creates a degree of mixing that maintains solids or heavy molecular weight solutes in a manageable suspension without fouling the heat transfer surface. Mass diffusivities in ATFEs can be increased by 1,000 to 10,000 times over nonagitated designs. To further promote separation, the unit is usually operated under vacuum conditions which permits lower temperature processing of thermally unstable mixtures.

Finally, thermodynamic properties of the waste and ATFE operating pressure set a limit on ultimate recovery imposed by vapor-liquid equilibrium constraints. Material will boil when its vapor pressure reaches the operating pressure of the ATFE. Waste vapor pressure for miscible fluids is equal to the sum of the partial pressure of each volatile species. Partial

TABLE 19. ATFE- WASTE CHARACTERIZATION

Facility	Source of Waste	Major Solvent Components	RCRA Waste Code
J	Oil degreasing	Perchloroethylene	F001
J	Oil degreasing	Trichloroethylene 1,1,1-trichloroethane Perchloroethylene	F001
J	Various generators	1,1,1-trichloroethane	F001
K	Ink printing	Ethyl acetate Ethanol N-propyl acetate	F003/ F005
K	Paper industry	Heptane Xylene Toluene Methyl ethyl ketone	F003/ F005
K	Various generators	Trichloroethylene	F001
L	Paint and lacquer	Toluene Methyl ethyl ketone Acetone Isopropanol	F003/ F005

pressure of each component is, in turn, equal to its molar concentration multiplied by the pure component vapor pressure and a constant which is dependent on the ideality of the solution. Thus, operating pressure and partial pressure determine the minimum attainable (i.e., equilibrium) concentration of each volatile species in solution. High separation efficiency will be associated with low system pressure, high pure component vapor pressure, high activity coefficient, high Henry's Law constant, and low solubility (solubility decreases with temperature). Theoretically, very high separations can be achieved for highly volatile compounds in systems of low liquid phase miscibility.

Ultimate recovery will depend on the extent to which equilibrium is achieved, which will be limited by diffusive resistance to mass transfer and residence time in the system. For viscous wastes, economical recovery is limited by waste viscosity as a result of decreasing diffusivity of volatile compounds through the waste as its viscosity increases. As diffusivity decreases, resistance to overall mass transfer into the gas phase increases. However, this effect is less pronounced in ATFEs relative to other evaporator designs due to high turbulence and large exposed waste surface area.

Residual solvent concentrations below 1,000 ppm have been routinely achieved and a concentration below 100 ppm can be achieved if conditions are optimal. However, except in unusual circumstances (e.g., immiscible fluids), the sole use of ATFE cannot be expected to yield residual concentrations of volatiles in the low ppm range.

Heated Screw Auger Evaporator

The heated screw auger is capable of treating non-pumpable wastes such as drum heels, viscous still bottoms and any other high solids content waste (e.g. soils). The process is similar to thin film evaporation. The waste is shred prior to entering the unit and then is transported through an air-locked cylinder by a screw auger. As in the operation of the thin film evaporator, the solvent removal in this process occurs as the waste is heated during its path down a cylinder which is heated with a hot air jacket. The turbulence of the solid waste facilitates efficient heat and mass transfer and creates a degree of mixing that diminishes fouling on the heat transfer surface.

The effect of thermodynamic properties on ultimate recovery of solvents using the heated screw auger is analagous to thin film evaporation theory and is explained in the theory section for the thin film evaporator.

FACILITY J

Process Description

Figure 7 presents a schematic of the top and side views of the thin film evaporator used at Facility J. Waste solvents, contaminated with oil from degreasing operations (F001), to be recycled are pumped from drums using a gear pump. The rate at which waste is introduced to the evaporator

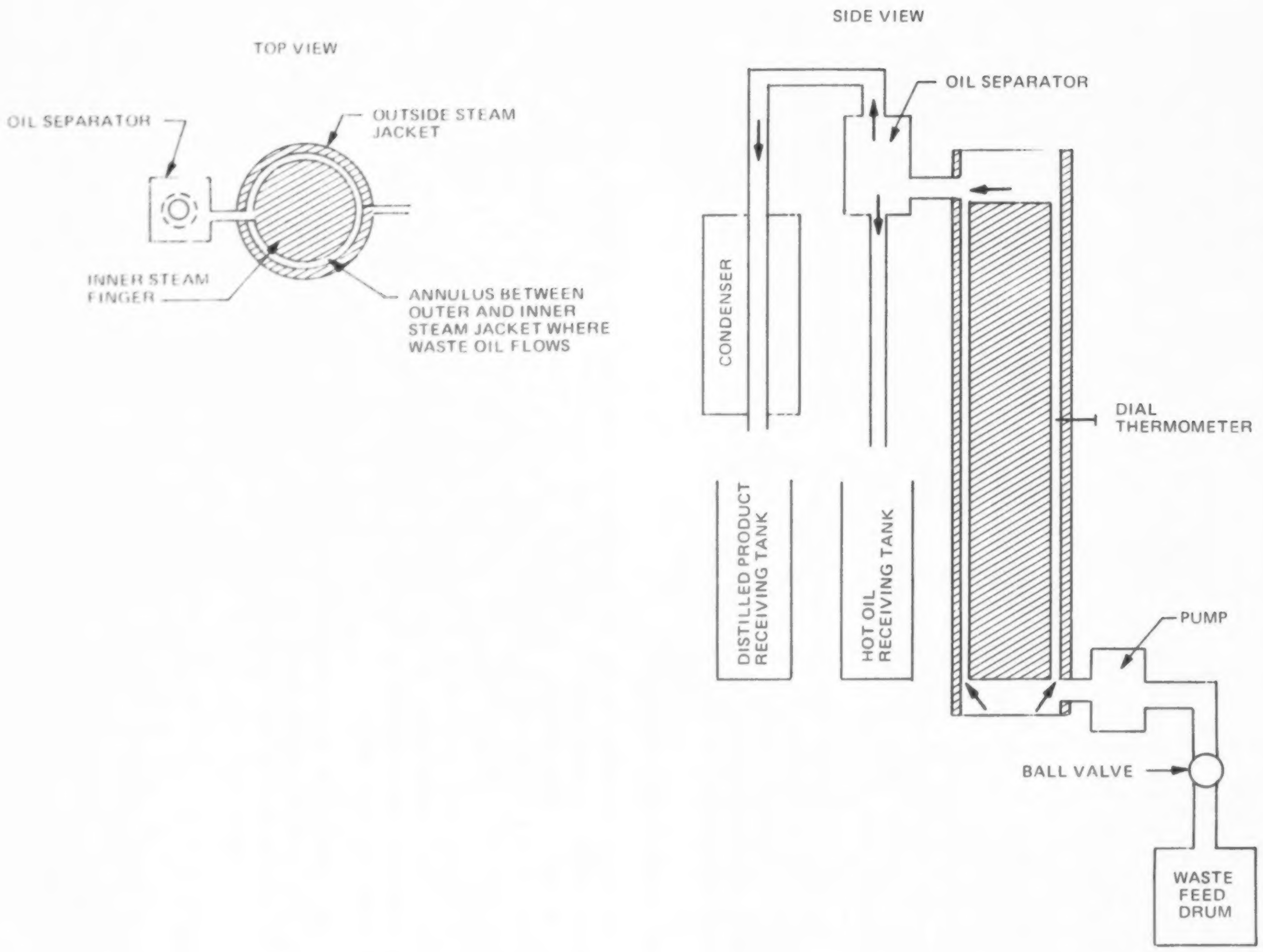
TOP VIEW
OIL SEPARATOR
OUTSIDE STEAM JACKET
INNER STEAM FINGER
ANNULUS BETWEEN OUTER AND INNER STEAM JACKET WHERE WASTE OIL FLOWS
SIDE VIEW
OIL SEPARATOR
CONDENSER
DISTILLED PRODUCT RECEIVING TANK
HOT OIL RECEIVING TANK
DIAL THERMOMETER
PUMP
BALL VALVE
WASTE FEED DRUM

Figure 7. Thin film evaporator at Facility J.

is controlled using a ball valve. The feed rate varies from 1 to 3 gallons per minute. Wastes are pumped through the gear pump into the bottom of the thin film evaporator (TFE) and pushed up through a small annular space between an outside steam jacket and an inner steam finger. This creates a thin film of waste material which can be heated quickly. The operating temperature of the unit is dependent on the specific solvent being separated from the oil. A high boiling solvent such as perchloroethylene requires temperatures in excess of 300° F. Other solvents such as trichloroethylene or 1,1,1-trichloroethane can be treated at temperatures well below 300° F. The temperature is monitored by a dial thermometer located on the side of the unit.

When the waste oil reaches the top of the evaporator, it spills over into the separator. Solvent vapors rise from the top of the oil separator, pass through a condenser and are discharged to an open tank. As this tank is filled, the contents are removed and placed in drums to be returned or sold to a customer. Hot oil discharges from the bottom of the separator and is collected in an open tank. As this tank fills up, the contents are drummed. At a later date, the oil is added to a large tank in the warehouse area which contains recycled nonhalogenated solvents which were generated by another unit at the facility. The contents are mixed and then allowed to settle. The mixture is then sold to a cement kiln or another fuel outlet as a low-grade supplemental fuel. Following initial operator-assisted startup, the TFE can run continuously with little supervision. Typically, the unit is shut down at the conclusion of a shift or a batch and is usually not cleaned between runs.

The TFE used at Facility J is distinctly different from most other thin film evaporators used in the recycling industry. Whereas most thin film evaporators introduce wastes at the top of the unit, this facility introduces waste at the bottom. The unit has no moving parts while most conventional ATFE units use some type of rotary wiper blade to distribute incoming material. Movement of the waste through the evaporator is accomplished with a pump.

Typically, a thin film evaporator is used to recycle a contaminated solvent when the percentage of solvent to be recovered from the waste is high. Solvent is evaporated from the waste and recovered, while a still bottom results containing a small residual solvent level. The amount of solvent remaining depends on overhead product purity specifications and desired disposition of the still bottoms stream.

When a solvent waste is classified as F001 (i.e., a halogenated solvent used in degreasing operation), Facility J will purchase the bottoms of the waste stream after it has been recycled. These bottoms will be directed through the thin film evaporator to recover any solvent remaining in the oil. The oil will be fuel blended as discussed previously. Facility J will also reprocess any still bottoms recovered from other stills used at the facility.

The TFE unit usually processes about 1,200 gallons of oily still bottoms per 8-hour shift. Assuming that there are 250 working days a year, the unit can process about 300,000 gallons (just under 5,500 drums) per year. This capacity could be significantly increased with the addition of a second or third shift and/or weekend operation. Recently, the facility added a second shift to process a backlog of drums which had stockpiled at the facility.

Sampling Program

The thin film evaporator process at Facility J was sampled on October 28 and 30 and November 1, 1985.

Waste feed to the thin film evaporator was pumped directly from drums into the unit. To ensure that a representative sample was obtained, at least six drums of waste were run through the unit. To address any possible phase separation within the drum, waste was grab-sampled at three stages: 1) shortly after a drum was introduced to the unit, 2) when the drum was about half empty, and 3) when the drum was nearly empty. Grab samples were taken of the solvent product and separated oil 3 minutes after every waste feed grab since the residence time of the waste in the unit is approximately 3 minutes. Taking grab samples of solvent product and separated oil 3 minutes after sampling the waste feed ensured that these samples would be the appropriate output products of the waste feed material.

The waste feed samples were collected from a valve upstream of the gear pump. Grabs were made with a pitcher and were then apportioned to the appropriate sample containers. The separated oil and solvent product came out of open discharge tubes. A large stainless steel ladle was used to collect the solvent product. Separated oil was collected using a stainless steel pitcher. The separated oil being discharged was extremely hot. To avoid problems with breakage of the glass sample containers, the pitcher of hot separated oil was placed in an ice bath for a few minutes before the oil was apportioned to sample containers.

Grab samples were composited differently for all three runs. Six drums of the waste were processed during each of the three sampling runs. During run one, composite samples were collected during the processing of each set of two drums, therefore there were a total of three waste samples to be analyzed. During run two, all grab samples were composited and then divided into thirds, thus the composite sample could be analyzed in triplicate. During run three, all grabs were composited to make one sample. In each case, the same procedure applied to waste feed, separated oil, and solvent product sampling locations.

Data

The analytical data for the three TFE runs at Facility J are presented in Table 20.

TABLE 20. FACILITY J ANALYTICAL RESULTS

POLLUTANT	UNITS	RUN 1 WASTE FEED *	 DISTILLATE*	 STILL BOTTOMS *	RUN 2 WASTE FEED *	 DISTILLATE *	 STILL BOTTOMS *	RUN 3 WASTE FEED	 DISTILLATE	 STILL BOTTOMS
ORGANICS						U / L				
perchloroethylene	wt %	37	87	25	0.105	NS NS	108 ppm	33	84	26
trichloroethylene	wt %	0.65	ND	ND	1.7	0.061 0.063	0.086	ND	ND	ND
1,1,1-trichloroethane	wt %	0.31	ND	ND	0.24	0.20 0.18	57 ppm	ND	ND	ND
methylene chloride	wt %	ND	0.65	ND	ND	ND ND	ND	ND	ND	ND
METALS										
silver	ug/g	<0.23	<0.003	<0.24	7.1	0.02	18	<0.24	<0.003	<0.24
arsenic	ug/g	<0.94	<0.01	<0.97	<0.98	<0.05	<0.98	<0.95	<0.01	<0.97
beryllium	ug/g	0.033	<0.0005	0.017	0.082	0.002	0.157	0.026	0.0002	0.044
cadmium	ug/g	0.086	<0.0006	0.137	9.14	0.033	18	0.143	<0.0005	0.218
chromium	ug/g	<1.15	<0.013	<1.02	15.6	<0.004	38.7	0.357	<0.0008	0.558
copper	ug/g	<5.9	0.041	8.2	570	5.1	1358	3.6	0.05	4.1
nickel	ug/g	<0.316	<0.006	0.347	12.2	0.017	27.3	<0.143	0.003	0.291
lead	ug/g	<4.84	0.015	8.6	46	0.56	57	3.3	<0.005	1.5
antimony	ug/g	<1.2	<0.013	<1.2	<1.3	<0.06	1.7	<1.2	<0.015	<1.2
selenium	ug/g	<0.94	<0.008	<0.97	<0.98	<0.05	<0.96	<0.95	<0.01	<0.97
thallium	ug/g	<0.71	<0.009	<0.73	<0.89	<0.04	<0.73	<0.71	<0.008	<0.7
zinc	ug/g	9.44	0.029	13	320	6.55	734	8.75	0.015	12.5
OTHER ANALYSES			U/L						U/L	
density	g/ml	1.1	1.0/1.6	0.97	0.95	1.0	0.91	1.0	1.0/1.6	0.98
heat content	btu/lb	13,633	NS	15,605	10,287	NS	18,520	13,580	NS	15,230
viscosity	ssu @ 100 F	40	NS	47	242	NS	42.3 **	41	NS	46
% chlorine	wt %	23.9	NS	16	2.15	NS	1.5	23.6	NS	16.0
% carbon	wt %	57.3	NS	68.4	52.2	NS	78.9	58.3	NS	66.5
% hydrogen	wt %	8.02	NS	9.8	32.1	NS	12.1	8.06	NS	9.6
% nitrogen	wt %	0.02	NS	0.02	0.04	NS	0.07	0.01	NS	0.05
% oxygen	wt %	10.7	NS	5.6	34.1	NS	5.73	10	NS	7.8
% sulfur	wt %	0.05	NS	0.09	0.3	NS	0.61	0.06	NS	0.08
% ash	wt %	0.03	NS	0.05	0.52	NS	1.2	0.04	NS	0.03
% water	wt %	0.31	0.04	0.03	32/9.2	NS	0.11	0.31	0.04	0.03
total chloride	ug/g	239,000	437,333	170,333	21,500	11.6 ug/l	15,030	236,000	548,000	160,000

ND = Not Detected
NS = Not Sampled
U/L = Upper phase / Lower phase
* = Average of three samples
** = SSF @122 degree F
NM = Not Measured
SSU = Seconds Saybolt Universal (Viscosity Units)
SSF = Seconds Saybolt Furol (Viscosity Units)

Waste Characterization --

The waste stream for run 1 consisted of perchloroethylene and oil from a degreasing operation. As expected perchloroethylene was the major component at a concentration of 37 weight percent. Zinc, at a concentration of 9.44 μg/g, was the primary metal detected in the waste stream. The feed rate to the evaporator was 2.2 gal/min and the operating temperature was 300°F. During run 3 the same type of waste stream was treated as in run 1 but the feed rate was 1.0 gal/min. Again the primary component was perchloroethylene at 33 weight percent and the most concentrated metal was zinc at 8.75 μg/g. The total chloride concentration in the waste stream for run 1 and 3 were 239,000 and 236,000 μg/g respectively.

During run 2 the thin film evaporator treated a mixture of 1,1,1-trichloroethane, trichloroethylene and oil from a degreasing operation. The evaporator was operated at a feed rate of 1.4 gal/min and a temperature of 250° F. The concentration of trichloroethylene in the waste stream was 1.7 weight percent and the stream was 52.2 percent carbon thus one or more compounds is unidentified. Metals concentrations detected in this waste stream were significantly higher than for runs 1 and 3. Copper, zinc, and lead were measured at 570, 320, and 46 μg/g respectively. The total chloride concentration was 21,500 μg/g.

Product Characterization --

For the perchloroethylene waste streams (run 1 and 3) the thin film evaporator produced a distillate with 84-87 weight percent perchloroethylene and 0.04 weight percent water.

The trichloroethylene waste stream (run 2) produced a distillate product containing 0.061 weight percent trichloroethylene and 0.20 weight percent 1,1,1-trichloroethane. The distillate also contained 6.5 μg/g of zinc and 5.1 μg/g of copper. Of the metals detected, only cadmium and chromium exceeded levels which are normally found in used oils according to a study by EPA (EPA /530-SW-013, November 1984) and thus the potential for further environmental problems exist.

Residue Characterization --

The solvent perchloroethylene was reduced to 26 weight percent in the bottoms product for runs 1 and 3. The heat content of the bottoms for the two runs was approximately 15,500 Btu/lb an increase of 2000 Btu/lb over the fuel value of the feed stream. The chlorine content in the bottoms was 16.0 percent Cl for both runs.

The concentration of trichloroethylene was reduced to 0.086 weight percent in the bottoms product of run 2 and the heat content increased from 10,300 to 18,500 Btu/lb. The chlorine content of the bottoms was also reduced to 1.5 percent Cl.

Evaluation of Effectiveness

For runs 1 and 3, in which the waste processed came from the same generator, Facility J's analytical laboratory failed to correctly characterize the waste as containing more than 20 percent perchloroethylene. Preliminary analyses by Facility J indicated a concentration of about 3 percent perchloroethylene. When the halogenated solvent content of an oily still bottom is high, Facility J will first process the oil through the roughing still to reduce the level of halogenated solvent to a concentration below 18 percent. Because Facility J determined (incorrectly) that the stream contained only 3 percent perchloroethylene, they introduced the waste directly into the thin film evaporator. Preliminary treatment on the roughing still was not done. Subsequent analysis of the waste feed stream indicated that the concentration of perchloroethylene averaged about 35 percent. This concentration was confirmed by Gas Chromatograph/Flame Ionization Detector (GC/FID) and GC/MS analysis. Based on these results, Facility J processed a waste stream through the thin film evaporator that should have been subjected to the preliminary treatment step.

The analytical data for runs 1 and 3 show a reduction in solvent content from about 35 percent to about 26 percent in the waste feed and separated oil, respectively. Facility J plant personnel stated that because perchloroethylene has a relatively high boiling point (b.p. = 250° F), it is one of the hardest halogenated solvents to remove from the still bottoms. Results indicate that the reduction of perchloroethylene was between 20 to 30 percent. A higher solvent removal efficiency would be expected if the waste feed stream had a much lower perchloroethylene content than it did since the TFE normally treats waste streams containing less than 18 percent halogenated solvent.

Data for run 2 indicate that the thin film evaporator is effective in removing halogenated solvents from oily still bottoms. During the run, a waste containing trichloroethylene, 1,1,1-trichloroethane and perchloroethylene was processed. Unlike the other two runs on a perchloroethylene containing waste, the solvent content of this waste was in the range that the thin film evaporator was designed to treat (2 to 18 percent solvent). During run 2, all three chlorinated solvents showed a 90 percent or greater reduction in the oily still bottoms.

Removing halogenated solvents increases the fuel value of oily still bottoms. During all three runs, an increase in fuel value from waste feed to separated oil bottoms was observed. Most halogenated solvents are nonflammable and therefore, would compromise the fuel value of the bottoms. However, the increase in fuel value of the bottoms cannot be attributed just to solvent removal. Karl Fischer tests show a reduction in water content from waste feed to separated oil. During runs 1 and 3, the water content was reduced from 0.31 percent in the waste feed to 0.03 in the bottoms. The amount of water in this stream is low and probably does not impact the fuel value appreciably. In run 2, two aliquots were taken from the waste feed sample to analyze for water. An aliquot from near the top contained 32.0 percent water and an aliquot from near the bottom was 9.2 percent water. In the oily still bottoms, the percentage of water was 0.11 percent. During run 2, water removal from the waste feed was the primary contributor to the increase in fuel value.

Although the heat content of the wastes for all three runs increased, the chlorine concentration in the bottoms did not fall below 3 percent Cl for all three runs. Facility J's operations are geared, in part, to the state requirements which do not allow wastes containing more than 3 percent organic chlorine by weight to be burned in cement kilns. Runs 1 and 3 only achieved a reduction to 16 percent chlorine in the bottoms thus the bottoms are poor candidates for use as a supplemental fuel. During run 2, the chlorine content was reduced to 1.5% in the bottoms thus this run met the criteria for chlorine content. In fact, before the waste was processed, the chlorine content was only 2.15% which is already below the criteria of 3% organic chlorine.

The thin film evaporator has the effect of concentrating metals in oily still bottoms. During runs 1 and 3, low levels of chromium, copper, lead and zinc were found in the waste feed stream. With the exception of chromium, the concentration of these metals was higher in the separated oil bottoms than in the waste feed. During run 2, low levels of silver, cadmium, chromium, nickel and higher levels of lead, copper, and zinc were found in the waste feed stream. With the exception of lead, all of these metals were higher in the separated oil bottoms than in the waste feed. The ultimate use for these bottoms is as a low grade fuel. If the concentration of metals in the stream is high, the potential for further environmental problems exists. In run 2, the levels of cadmium and chromium in the separated oil exceeded levels which are normally found in used oils according to a study by the EPA. From the available information M&E was unable to ascertain the fate of the separated oil bottoms.

Residue Management

The hot oil bottoms are discharged from the bottom of the oil separator and collected in an open tank. As this tank fills up, the contents are drummed. At a later date the oil is added to a large tank in the warehouse area which contains recycled nonhalogenated solvents generated by another treatment unit at the facility. The contents are mixed and then allowed to settle. The mixture is then sold to a cement kiln or another fuel outlet as a low-grade supplemental fuel.

FACILITY K

Process Description - ATFE

Facility K treats both halogenated and nonhalogenated solvents with a Luwa thin film evaporator.

The operation of the unit is fairly simple: waste solvents are pumped from a large holding tank or drum to the top of the evaporator. The feed line contains quick connects so that changing the feed to the unit is a fairly simple task. During the test period, the feed rate varied from 1.3 to 3.8 gallons/minute.

The Luwa thin film evaporator contains an internal stainless steel rotary blade which spins at approximately 385 revolutions/minute. As waste solvent is introduced at the top of the unit, the solvent flows by gravity over the rotary blade and internal wall surface. The rotary blade distributes the waste solvent evenly over the internal heated wall. The clearance between the rotary blade and the wall is 0.008 in. (0.20 mm). The unit has a heated surface area of 43 square feet. The internal cylindrical wall is heated by a hot oil jacket surrounding the thin film evaporator. Facility K chose to heat the unit with hot oil instead of steam so that they could operate the unit over a greater temperature range. In addition, the thin film evaporator is operated under vacuum to enhance the distillation process. The vacuum is pulled from the top of the distillate receiving tank.

As the waste solvent heats up during its flow over the internal wall, solvent vapors are given off and pass through the top of the unit. The vapors are vented through a mist entrainer prior to going through a condenser. The resulting distillate is collected in an enclosed 10-gallon receiving tank. The receiving tank is equipped with a liquid level control valve. This valve allows portions of the collected distillate to be pumped from the bottom of the receiving tank to a holding tank or a drum without having to release the system vacuum. The product distribution line located immediately downstream of the product pump is equipped with quick connects to allow recovered solvent to be pumped to different holding tanks. Still bottoms discharged to the bottom of the evaporator are pumped into a receiving drum before being transferred to a storage tank. The still bottoms pump is connected to the bottom of the evaporator with flexible tubing since rotation of the rotary blade inside the unit causes vibration. The thin film evaporator is equipped with a recirculation line such that the still bottoms can be recycled through the unit a second time if desired. Facility K seldom exercises this option, however. No still bottoms were recirculated through the evaporator during the test period.

During the first two runs, the facility was trying out a new Black Mar vane pump to transfer the still bottoms to the receiving drum. The variety of solvent wastes and the physical characteristics of the still bottoms tends to wear out the pump quickly. Problems were experienced with the new pump, however. In overcoming the vacuum in the Luwa, the evaporator could only process waste solvent at about one-third the normal rate. Following the end of the second run, the facility replaced the Black Mar pump with a new Viking pump. This change allowed the unit to process waste solvent at the normal rate. The TFE was operated at steady state in either case so the results should be representative of the TFE's effectiveness.

During a run, still bottoms regularly collect in both the base of the Luwa and the flexible tubing immediately upstream of the pump. When this happens, the operator releases the vacuum slightly and increases the feed rate to the evaporator. These actions have the effect of causing a quick waste solvent wash of the Luwa and flushing the viscous still bottoms through the bottoms pump.

Facility K tries to achieve as high a reclaimed solvent yield as possible. However, to operate the unit properly, the still bottoms must remain pumpable. Consequently, to satisfy this requirement, a small percentage of solvent is allowed to remain in the bottoms. In addition, for nonhalogenated bottoms, the residual solvent helps to improve the fuel value. On occasion, the facility will recycle the bottoms through the evaporator following low yield runs. Facility K does not landfill any of the bottoms generated by the Luwa. These materials are shipped off-site to a fuel-blending facility or directed to a cement kiln to be used as a supplemental fuel. Figure 8 presents a schematic diagram of the thin film evaporator.

Sampling Program

Sampling of the agitated thin film evaporator at Facility K took place on December 3, 4, and 5, 1985.

Waste feed to the ATFE was pumped directly from storage tanks to the unit. During each day of sampling, the ATFE was allowed to operate for a period of 30 minutes to an hour before sampling began. This delay ensured that the unit had achieved normal steady-state operating conditions. Each of the three sampling runs were 4 hours in length. Once at the beginning and every half hour thereafter, a grab sample was taken of the waste feed, still bottoms and overhead product. Waste feed samples were obtained from a valve upstream of the waste feed pump. Still bottoms were collected from an open discharge pipe to the receiving drum downstream of the recirculation pump. Overhead product samples were collected from a valve downstream of the condenser. Nonhalogenated solvent wastes were processed during two of the three runs. The third run was a halogenated waste (trichloroethylene). During this run, waste feed, overhead product and still bottom samples were collected in triplicate. The purpose of triplicate sampling was to determine sampling and analytical precision and to verify whether or not sample contamination took place during transport back to the analytical laboratory. Sampling took place all three days without incident.

Data

Results for the three waste streams that were treated during the sampling episode are presented in Table 21.

Waste Characterization --

During the sampling episode, two of the waste streams were nonhalogenated waste solvents (F003 and F005) and one of the waste streams was a halogenated waste solvent (F001, trichloroethylene). For run 1 the primary constituent in the waste stream was trichloroethylene at 103 weight percent. In order to quantitate this compound, it was necessary to dilute the sample several orders of magnitude so that a response could be recorded that did not saturate the GC/FID. Slight differences in response get magnified when

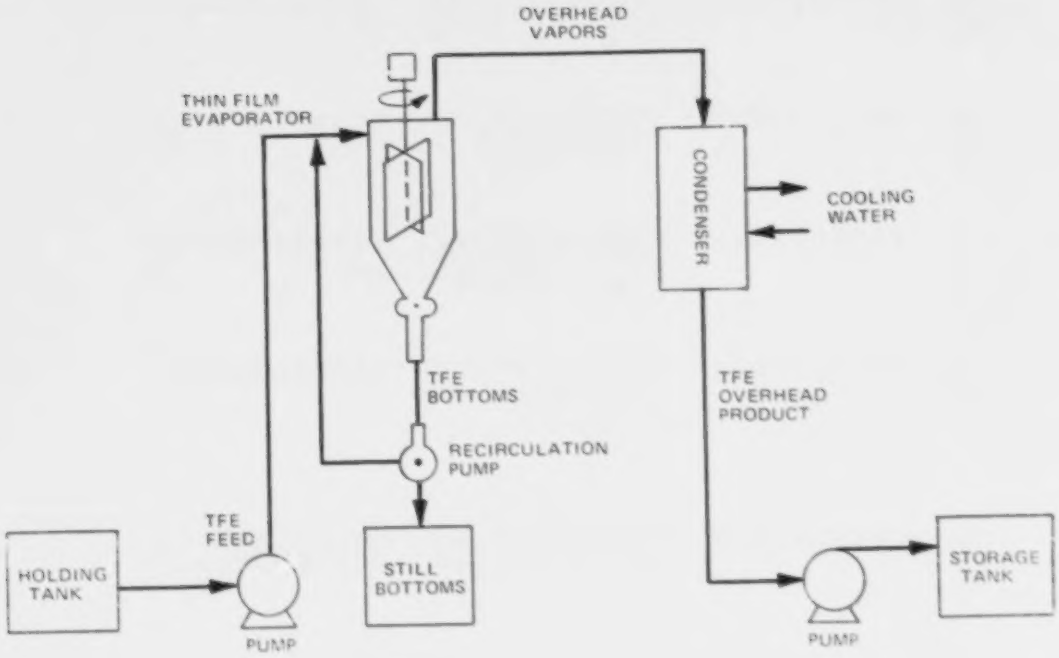
OVERHEAD VAPORS
THIN FILM EVAPORATOR
CONDENSER
COOLING WATER
TFE BOTTOMS
TFE OVERHEAD PRODUCT
RECIRCULATION PUMP
TFE FEED
HOLDING TANK
PUMP
STILL BOTTOMS
STORAGE TANK
PUMP

Figure 8. Thin film evaporator process schematic - Facility K.

TABLE 21. FACILITY K - THIN FILM EVAPORATOR ANALYTICAL RESULTS

POLLUTANT	UNITS	RUN 1			RUN 2			RUN 3		
		WASTE FEED	DISTILLATE	STILL BOTTOMS	WASTE FEED	DISTILLATE	STILL BOTTOMS	WASTE FEED	DISTILLATE	STILL BOTTOMS
ORGANIC										
trichloroethylene	wt %	103	110	75	ND	ND	ND	ND	ND	ND
ethanol	wt %	ND	ND	ND	24	30	20	ND	ND	ND
n-propanol	wt %	ND	ND	ND	7.7	4.9	14	ND	ND	ND
ethyl acetate	wt %	ND	ND	ND	24	39	8.3	ND	ND	ND
n-propyl acetate	wt %	ND	ND	ND	9.8	10	11	ND	ND	ND
2-butanone	wt %	ND	ND	ND	ND	ND	ND	1.8	2.6	<0.2
toluene	wt %	ND	ND	ND	ND	ND	ND	50	50	47
heptanes	wt %	ND	ND	ND	ND	ND	ND	15	21	3.9
ethyl benzene	wt %	ND	ND	ND	ND	ND	ND	1.7	0.8	3.2
xylene	wt %	ND	ND	ND	ND	ND	ND	6.9	2.5	14.7
METALS										
silver	ug/l	0.09	<0.002	0.48	8.9	<0.003	17.0	0.01	<0.003	<0.13
arsenic	ug/l	<0.03	<0.007	<0.50	<1.0	<0.012	<1.0	<0.05	<0.013	<0.5
beryllium	ug/l	<0.0004	<0.0001	<0.006	<0.013	<0.0002	<0.013	<0.0006	<0.0002	<0.006
cadmium	ug/l	0.188	0.0004	1.47	<0.05	<0.0006	0.170	0.006	<0.0007	<0.026
chromium	ug/l	1.86	0.0017	10.41	25.8	<0.001	61.9	1.24	0.002	1.91
copper	ug/l	2.23	0.0143	23	79.0	0.160	260.0	0.44	<0.0007	<0.26
nickel	ug/l	0.225	0.0043	1.16	0.52	<0.002	1.2	0.052	0.004	<0.077
lead	ug/l	2.0	<0.004	5.47	25.0	<0.006	46.0	3.5	<0.007	15
antimony	ug/l	<0.04	<0.009	<0.64	<1.3	<0.015	<1.3	<0.06	<0.016	<0.64
selenium	ug/l	<0.03	<0.007	<0.51	<1.0	<0.012	<1.0	<0.05	<0.013	<0.50
thallium	ug/l	<0.02	<0.005	<0.40	<0.75	<0.009	<0.75	<0.04	<0.010	<0.40
zinc	ug/l	5.88	0.0047	58.4	13.3	0.011	31.3	0.492	0.004	1.94
OTHER ANALYSIS										
percent solids	wt %	1.9	0.008	15.2	11.8	0.035	36.5	7.4	0.033	23.7
density	g/ml	1.43	1.43	1.34	0.86	0.85	1.07	0.79	0.78	0.88
heat content	btu/lb	NM	NM	6603	NM	NM	11970	NM	NM	16690
viscosity	sus @ 100 F	NM	NM	33	NM	NM	135	NM	NM	45
% carbon	wt %	NM	NM	29.5	NM	NM	b	NM	NM	73.0
% hydrogen	wt %	NM	NM	3.55	NM	NM	b	NM	NM	9.48
% nitrogen	wt %	NM	NM	0.07	NM	NM	1.16	NM	NM	0.12
% oxygen	wt %	NM	NM	0.6	NM	NM	b	NM	NM	16.3
% sulfur	wt %	NM	NM	0.03	NM	NM	0.24	NM	NM	0.01
% chlorine	wt %	NM	NM	66.1	NM	NM	c	NM	NM	c
% ash	wt %	NM	NM	0.17	NM	NM	4.50	NM	NM	1.1
Volume yield per liter of waste feed	liters	1	0.698	0.302	1	0.376	0.624	1	0.458	0.542

b = Sample exploded in combustion chamber.
c = These runs were on nonhalogenated stream. Therefore, a total chloride analysis was not done.
ND = Not Detected
NM = Not Measured

calculating concentration in the sample and consequently explains why an average concentration of greater than one hundred percent by weight trichloroethylene was observed. The feed rate to the thin film evaporator during run 1 was 1.3 gal/min and the hot oil entering and exiting the heating jacket was approximately 300° F. The solids content in the waste was 1.9 percent.

The nonhalogenated waste stream treated during run 2 was generated by an ink printing operation and contained ethanol, ethyl acetate, n-propyl acetate and n-propanol. Ethanol and ethyl acetate were each measured at 24 percent by weight in the feed stream. Significant levels of silver, chromium, copper, lead and zinc were also measured. The waste stream was 12 percent by weight solids and was fed at an average rate of 2.3 gal/min to the thin film evaporator. The hot oil entering the heating jacket was 318° F and the exiting temperature was 307° F.

Run 3 was also a nonhalogenated waste stream generated from two paper companies and contained methyl ethyl ketone, heptane and toluene. Methyl ethyl ketone comprised 50 percent by weight of the influent stream. The solids content was 7.4 weight percent. The feed rate to the TFE for run 3 averaged 3.8 gal/min. The hot oil entering the heating jacket was 327° F and the exiting temperature was 317° F.

Product Characterization --

The distillates produced during all three runs were fairly clean. The waste feed for all three runs appeared either black, brown or blue and was quite murky whereas the distillate was a very clear liquid. The levels of solids detected can probably be attributed to experimental error or minor equipment contamination.

The distillate for run 1 contained 110 weight percent trichloroethylene. This concentration of greater than 100 weight percent can be attributed to discrepancies resulting from dilution of samples to quantitate trichloroethylene just as in the case of the influent waste stream.

Run 2 produced a distillate comprised of 39 percent ethyl acetate, 30 percent ethanol, 10 percent n-propyl acetate and 5 percent n-propanol.

The major component recovered in the distillate of run 3 was toluene at 50 percent by weight of the product which is the same concentration of toluene detected in the influent waste stream.

Residue Characterization --

The level of trichloroethylene in the bottoms from the halogenated waste stream run (run 1) was reduced to 75 weight percent. Metals were concentrated in the still bottoms with zinc and chromium being measured at 58 and 10 µg/l. The heat content of the bottoms was 6600 Btu/lb and the solids content of the residue was 15 percent by weight. The percent chlorine measured in the bottoms was 66 percent.

The still bottoms generated from operation of the TFE during run 2 showed a reduction in ethyl acetate to 8.3 weight percent but ethanol was only reduced to 20 weight percent. Metals again were concentrated in the still bottoms. Copper, at 260 μg/l was the primary metal detected but silver, chromium, lead, and zinc were also detected above 10 μg/l. The heating content of the bottoms is approximately 12,000 Btu/lb and the solids concentration was 36.5 percent.

Run 3 of the thin film evaporator only reduced the level of toluene from 50 percent in the influent to 47 percent in the bottoms product. The heat content was measured as 16,700 Btu/lb with a solids content of 23.7 percent. The only metal that had a concentration greater than 10 μg/l was lead which was measured at 15 μg/l. The EP toxicity test was not performed on the still bottom samples because they were predominantly liquid, contained fairly high levels of solvent and Facility K does not landfill these streams.

Evaluation of Effectiveness

Treatment of the halogenated waste during run 1 reduced the concentration of trichloroethylene from 103 weight percent in the waste fuel to 75 weight percent in the bottoms product. Since trichloroethylene is a nonflammable solvent, the appreciable quantity of trichloroethylene left in the bottoms limits its supplemental fuel value. The low heat content of the bottoms, 6,600 Btu/lb, reflects the presence of the nonflammable solvent. Even though the heat content is low it is sufficient for fuel blending. The problem arises with the high chlorine content of approximately 65 percent. Bottoms with a high chlorine content are not desirable for fuel blending due to the possibility of formation of HCl during combustion; the TFE did not produce a still bottoms desireable for fuel blending but was effective at reducing the waste volume and recovering organic solvent.

For run 2 the thin film evaporator reduced the percentage of ethyl acetate and ethanol in the nonhalogenated waste feed to 8.3 percent and 20 percent respectively in the still bottoms. During run 3 the TFE did reduce the percentage of heptane and n-propyl acetate but toluene, which accounted for 50 percent by weight of the waste feed, made up 47 percent by weight of the bottoms. Facility K normally allows a small percentage of solvent to remain in the bottoms to keep the bottoms pumpable. In the case of nonhalogenated bottoms, the residual solvent also helps to improve the fuel value. The analysis of the still bottoms for fuel parameters for runs 2 and 3 showed heat contents of approximately 12,000 and 17,000 Btu/lb respectively. The heat content values along with measurements of percent carbon, percent hydrogen, percent nitrogen, percent oxygen, and percent sulfur indicate that the bottoms from the nonhalogenated streams are suitable for use as a low grade fuel supplement.

The percent solids data for all runs show that any solid material present in the waste solvent feed is essentially removed from the distillate. The waste solvent samples collected were typically black, brown or blue, and heavily laden with suspended solids. Conversely, the distillate product samples were quite clear in appearance. The percent solids determination on the still bottoms stream showed a percent solids content of less than fifty percent. These results indicate that a fairly high percentage of solvents must remain in the still bottoms to keep them pumpable so that the unit will operate properly.

Metals were found in all waste feed streams. The TFE has the effect of concentrating the metals in the still bottoms stream. The waste feed stream from run 1 showed very low levels of chromium, copper, lead and zinc. Run 2 contained silver, chromium, copper, lead and zinc and run 3 contained chromium, lead and a lesser amount of zinc. The metals concentration in the still bottoms can have additional environmental concern other than landfilling given that this waste stream is used as a fuel supplement. Thought must be given to potential metal emissions from the subsequent burning of such material.

Residue Management

Facility K does not landfill any of the bottoms generated by the thin film evaporator. These materials are shipped offsite to a fuel-blending facility or directly to a cement kiln to be used as a supplemental fuel. On occasion, Facility K will recycle the bottoms through the evaporator following low yield runs.

Process Description - Heated Screw Auger Evaporation

Facility K has designed a screw auger unit which is capable of significantly reducing the solvent content of viscous still bottoms, drum heels or any waste containing a fairly high solids content. Facility officials consider the unit to still be in the pilot-scale stage. Figure 9 presents a schematic of the screw auger. Waste is fed to the unit manually, whereby an operator scoops the waste from the drum and places it in a device resembling a large meat grinder. The grinder shreds the waste before it enters the unit. The waste enters through an air-lock rotary valve at one end of a long (~20 feet) horizontal cylinder. A screw auger inside the cylinder transports the waste through the cylinder. Dolomite ($CaMg(CO_3)_2$) is added with the feed to prevent adhesion of the waste to the screw auger and to generate CO_2 to reduce internal explosions. The clearance between the screw auger and the outside wall is one-eighth of an inch. The cylinder is heated by a hot oil jacket which is capable of achieving an internal temperature of 550 deg F. The unit normally operates under 15 inches Hg vacuum. As the waste is heated during its path down the cylinder, solvent vapors are released. The vapors are collected by a manifold system and then pass through a mist entrainer and condenser. The condensed material is then collected in a receiving tank. The solid portion of the waste traverses the entire length of the cylinder and is discharged to a receiving drum. This drum is attached directly to the screw auger unit with a rubber seal in order to maintain the unit under vacuum. At the end of a run, the residual solids collection drum is disconnected from the unit. The residual solids discharged from the unit are considered hazardous under the RCRA regulations even though material tested in the past has passed EP-Toxicity tests. The facility has no interest at this point in time in attempting to have the

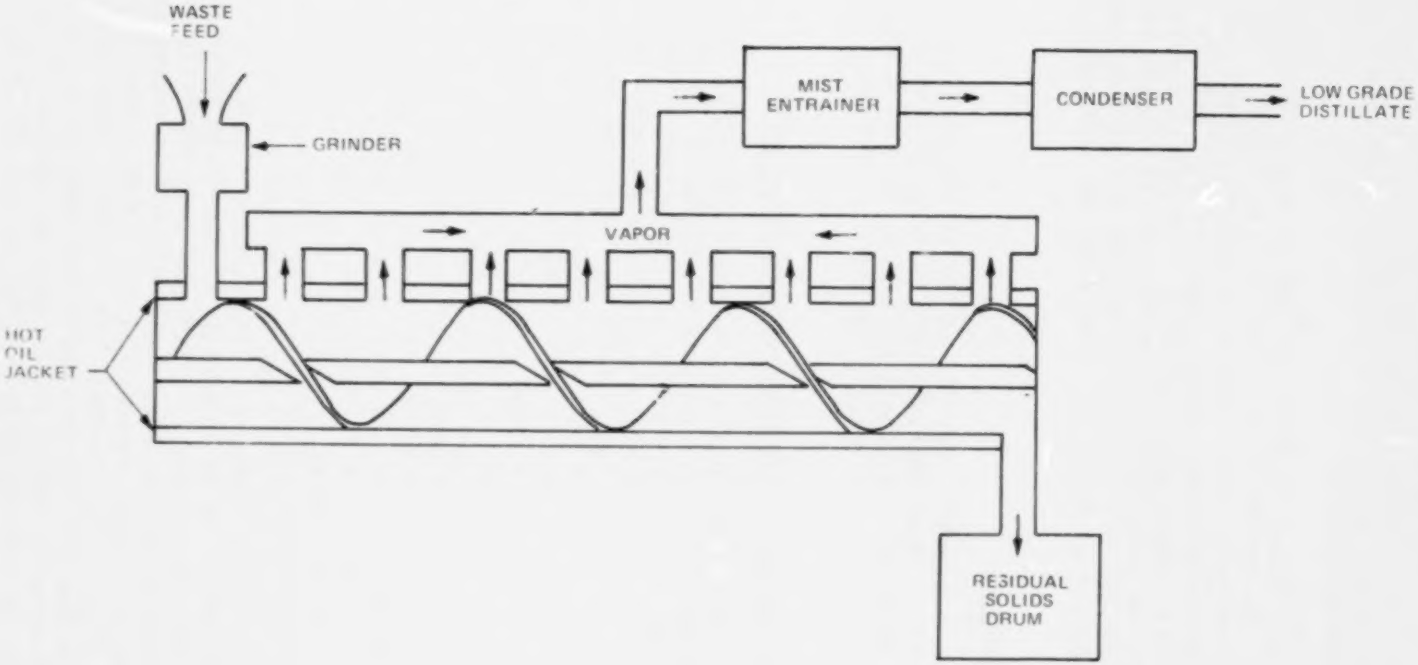
WASTE FEED
GRINDER
MIST ENTRAINER
CONDENSER
LOW GRADE DISTILLATE
VAPOR
HOT OIL JACKET
RESIDUAL SOLIDS DRUM

Figure 9. Screw auger process schematic - Facility K.

residual solids material delisted. Consequently, they seldom operate the screw auger unit. When they do operate the unit, however, it is for non-hazardous wastes. Residual solids that have been generated from processing hazardous wastes in the past have been blended with the Luwa still bottoms prior to being shipped off-site.

The screw auger unit is equipped with gages to measure the temperature of the hot oil in the jacket surrounding the unit and the condenser coolant, and a gage to measure the vacuum. For the single run monitored, the hot oil jacket temperature was 554° F, the coolant temperature was 50° F, and the vacuum was less than 10 inches of mercury.

Sampling Program

The heated screw auger was sampled on Thursday, December 5, 1985.

Four times during the run, a container was given to the operator who was feeding waste to the screw auger to collect a grab sample of the waste from the feed drum. All four of these grabs were composited to make one sample. Because of the long residence time in the cylindrical shell, a residual solids sample was collected once at the completion of the run when the receiving drum could be disconnected from the unit. Distillate grab samples were taken four times during the run from a valve upstream of the receiving tank. Each distillate grab sample was taken 15 minutes after a waste feed grab had been collected. The grab samples collected were composited together to make one sample. The distillate grab samples had to be taken quickly because the vacuum on the unit had to be shut off to collect the sample. The interruption in maintaining the unit under vacuum was not normal operation, but the small amount of time involved, less than one minute, should not have had any appreciable effect on the unit's overall performance.

Data

Table 22 presents the analytical results for waste feed, distillate and residual solids for Facility K's heated screw auger. EP toxicity results for the residual solids are presented in Table 23.

Waste Characterization --

The waste feed to the screw auger contained approximately 29 weight percent total solvent with xylene being the major solvent component at a concentration of 19 weight percent. Significant levels of metals were found in the waste stream. In particular, lead and chromium were measured at 8,800 and 2,170 μg/g. Arsenic, copper and zinc were also detected at levels greater than 50 μg/g.

Product Characterization --

The recovered distillate was comprised of two phases. The lower phase, comprising 90 percent of the distillate by volume, was primarily water that was contaminated with organic compounds. The sum of organic compounds for

TABLE 22. FACILITY K - HEATED SCREW AUGER ANALYTICAL DATA SUMMARY

	Waste Feed	Residual Solids	Distillate (Upper Phase)	Distillate (Lower Phase)
Principal Solvents (wt. %)				
Ethanol,	<0.02	<0.02	0.11	<0.2
Toluene,	4.3	0.15	6.2	0.08
Ethyl Benzene,	5.0	0.37	11	0.02
Xylene,	19.4	1.9	43	0.08
Sum,[a]	28.7	2.4	60.2	0.18
Metals (µg/g)				
Ag,	<1.0	<1.0	NS	NS
As,	52	86	NS	NS
Be,	<0.05	<0.05	NS	NS
Cd,	0.79	0.200	NS	NS
Cr,	2,170	3,470	NS	NS
Cu,	88	60	NS	NS
Ni,	17.6	35	NS	NS
Pb,	8,800	15,000	NS	NS
Sb,	<5.0	<5.0	NS	NS
Se,	<4.0	<4.0	NS	NS
Tl,	<3.0	<3.0	NS	NS
Zn,	352	490	NS	NS
Density, (g/ml)	-	-	0.81	1.0
Sample Volume, (%)	-	-	10	90
Heat Content, (Btu/lb)	14,780	7,420	-	-
% C	76.0	37.8	-	-
% H	9.92	4.56	-	-
% N	0.07	0.43	-	-
% O	0.54	0.74	-	-
% S	0.08	0.01	-	-
% Cl	0.06	0.06	-	-
% Ash	13.3	56.4	-	-
Total Chloride, (µg Cl/g)	612	638	-	-

[a] Less than values are not included in the sum.

TABLE 22. FACILITY K - HEATED SCREW AUGER ANALYTICAL DATA SUMMARY

	Waste Feed	Residual Solids	Distillate (Upper Phase)	Distillate (Lower Phase)
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Ethyl Benzene,	5.0	0.37	11	0.02
Xylene,	19.4	1.9	43	0.08
Sum,[a]	28.7	2.4	60.2	0.18
Metals (μg/g)				
Ag,	<1.0	<1.0	NS	NS
As,	52	86	NS	NS
Be,	<0.05	<0.05	NS	NS
Cd,	0.79	0.200	NS	NS
Cr,	2,170	3,470	NS	NS
Cu,	88	60	NS	NS
Ni,	17.6	35	NS	NS
Pb,	8,800	15,000	NS	NS
Sb,	<5.0	<5.0	NS	NS
Se,	<4.0	<4.0	NS	NS
Tl,	<3.0	<3.0	NS	NS
Zn,	352	490	NS	NS
Density, (g/ml)	-	-	0.81	1.0
Sample Volume, (%)	-	-	10	90
Heat Content, (Btu/lb)	14,780	7,420	-	-
% C	76.0	37.8	-	-
% H	9.92	4.56	-	-
% N	0.07	0.43	-	-
% O	0.54	0.74	-	-
% S	0.08	0.01	-	-
% Cl	0.06	0.06	-	-
% Ash	13.3	56.4	-	-
Total Chloride, (μg Cl/g)	612	638	-	-

[a] Less than values are not included in the sum.

TABLE 23. FACILITY K - SCREW AUGER EP TOXICITY RESULTS BY CURRENT AND PROPOSED PROCEDURES

	Current Procedure[a] (mg/l)	Proposed Procedure[b] (mg/l)	EPA Limits[b] (mg/l)
Volatile Organics			
Methylene Chloride	0.11	0.12	8.6
Acetone	1.7	0.91	c
4-Methyl-2-Pentanone	5.3	8.4	c
Toluene	3.3	5.2	14.4
Ethyl Benzene	0.94	5.9	c
Styrene	0.021	0.57	c
Xylene	7.4	15	c
Metals			
Ag	<0.005	<0.005	5.0
As	<0.02	<0.02	5.0
Ba	0.388	0.835	100.0
Cd	0.001	0.004	1.0
Cr	0.040	0.242	5.0
Hg	<0.002	<0.002	0.2
Pb	0.35	2.4	5.0
Se	<0.02	<0.02	1.0

[a] EP Toxicity Test Procedure, 46 FR 35247, July 7, 1981.

[b] Toxicity Characteristic Leaching Procedure (TCLP), FR 21685, June 13, 1986.

[c] None exist.

this phase was 0.18 weight percent. The upper phase consisted of organic solvents removed from the waste feed. Organics comprised 60.2 weight percent of the upper phase distillate and the primary component was xylene at 43 weight percent.

Residue Characterization --

The residual solids generated by the screw auger contained 2.4 percent total solvent. Significant levels of metals remained in the residue. Lead was measured at 15,000 μg/g and chromium was measured at 3,470 μg/g.

A fuel analysis of the residual solids indicates that the heat content is approximately 7,000 Btu/lb with a low chlorine content of 0.06 percent.

The EP toxicity test procedure and the Toxicity Characteristic Leaching Procedure were performed on the residual solids stream to detect the extent to which the metals and organics could leach out of the solids if landfilled. Even with the high concentration of metals in the residual solids the residue passed both testing procedures.

Evaluation of Effectiveness

The screw auger process is capable of achieving a significant reduction in waste solvent concentration. In the one run sampled, waste fed to the unit contained approximately 29 percent total solvent whereas the residual solids stream generated contained less than 3 percent total solvent. The recovered distillate was comprised of two phases, 90 percent of which was an aqueous phase which contained 0.18 percent by weight total solvent, and 10 percent of which was an organic phase which contained 60.2 percent by weight total solvent. Some water may have been originally present in the material fed to the screw auger, however, when chunks of dolomite were fed to the unit during operation water was also added. It is not known if this is standard procedure, or a consequence of the dolomite being frozen solid. In either case, the generation of a significant quantity of water contaminated with low concentrations of organics is not desirable.

The data resulting from the screw auger run suggest that some breakdown or recombination of the waste solvents may be taking place within the unit. The major organics in the waste feed were aromatic hydrocarbons (toluene, ethyl benzene and xylenes). In the lower water phase of the distillate some unidentified compounds were found. Based on the GC/MS spectrum of these compounds, they appear to be an aliphatic ketone and an ethoxy compound. This suggests that an oxidative reaction may have occurred in the unit.

A fuel analysis of the waste feed and residual solids stream shows that the solids stream has only one half the heating value of the feed stream. Apparently, the heating value of the waste feed is due to the residual solvents present in the waste. Facility K officials explained that they do not use the screw auger that often because the residual stream generated has a low heat value.

Significant levels of metal were found in both the waste feed and residual solids. The stripping process has the effect of concentrating metals in the residual solids stream. However, analysis of the EP toxicity and TCLP extracts for metals indicate that the residual solids stream would meet the EPA limits and be suitable for landfilling. Analysis of the extract of organics, similarly indicates that the solids residue stream would pass the TCLP test and be suitable for landfilling.

Residue Management

The residual solids discharged from the unit are considered hazardous under the RCRA regulations even if the materials pass the extraction tests. Currently, Facility K has no interest in attempting to have the residual solids material delisted. Consequently, they seldom operate the screw auger. The residual solids that have been generated from processing hazardous wastes in the past have been blended with still bottoms from a thin film evaporator process prior to being shipped offsite.

FACILITY L

Process Description

The thin film evaporator (TFE) used at Facility L is a horizontal tapered unit. The TFE is used solely for treatment of waste paint and lacquer thinners. The unit has 20 square feet (1.9 m^2) of heated surface area and generally operates at a rate of about 200 gph (12.6 Lpm). Heat is provided by a steam jacket and heating capacity is about 2 x 10^6 Btu/hr (2.1 x 10^9 J/hr). The temperature in the unit is manually controlled by regulating the steam pressure and/or waste feed rate. The TFE jacket is made of carbon steel while the shell is comprised of 304 stainless steel. The wiper blade assembly on the unit operates at a rotor speed of 300 rpm and a blade speed of 25-35 fps (7.6-10.7 mps). Clearance between the blade assembly and the vessel wall is variable (this is one of the advantages of this type of unit) and is normally set at one-eighth of an inch (0.32 cm).

The TFE is capable of operating under vacuum conditions up to 28 in. Hg (9,670 kg/m^2). The vacuum eductor system utilizes water from a nearby pond as the motive fluid. This pond also services the distillate product condenser.

Vapors exiting the TFE first pass through a 12-inch (30.5-cm) diameter demister section containing 6-inch (15.2-cm) demister pads and then through a 560-square foot (52-m^2) water-cooled condenser. Condensed product then flows into one of two small (30-gal. or 114-L) receiver tanks prior to discharge to a larger product storage tank. These two receiver tanks are alternated during the process such that the filled tank is isolated from the system and brought back to atmospheric pressure before pumping out the product while the other tank is connected to the process, brought under vacuum and begins collecting distillate product. Still bottoms are continuously collected in a 50-gallon (189-L) bottoms receiving vessel and subsequently pumped to large storage tanks. This still bottoms stream is shipped off-site for use as a supplemental fuel in asphalt kilns. The thin film evaporation process is illustrated in Figure 10.

Facility L uses the TFE to process waste paint and lacquer thinners that typically contain 2-20 percent dissolved paint resins plus suspended pigments and such nonhalogenated solvents as alcohols, ketones, esters, and aromatic hydrocarbons.

Sampling Program

Sampling of the TFE was done on Tuesday, August 19, 1986 during treatment of a waste paint and lacquer stream (RCRA designation F003/F005). According to the facility, toluene, methyl ethyl ketone, acetone, isopropanol, and water were the principal feed components. The unit processed 1,530 gallons of feed material over 6.75 hours and generated 830 gallons of distillate product and 688 gallons of still bottoms. The waste feed was a greyish brown homogeneous liquid while the product was clear and colorless. The still bottoms stream was a two-phased material, the top portion (80-90 percent by volume) being brown and the bottom being grey and semi-solid.

Data

The analytical results for samples taken during the operation of Facility L's thin film evaporator are presented in Table 24.

Waste Characterization --

The waste feed was a greyish brown homogeneous liquid. The major F001-F005 constituents found to be present in the waste feed were toluene, acetone and methyl ethyl ketone. These compounds were measured at levels of 220,000 mg/kg, 190,000 mg/kg and 180,000 mg/kg. Lesser amounts of isopropanol (76,000 mg/kg), xylenes (66,000 mg/kg) and ethyl benzene (22,000 mg/kg) were also found.

Low levels of RCRA metals were detected in the waste feed stream; titanium is reported only because it was found to be present at levels much higher than the other elements. Titantium would be expected in such a waste paint stream because titanium dioxide is a constituent of most pigments used in paints.

Product Characterization --

The distillate product stream (for which triplicate field samples were collected) was mainly toluene (240,000 mg/kg), methyl ethyl ketone (220,000 mg/kg) and acetone (180,000 mg/kg). On a mass basis, the distillation product yield from the feed material was 54 percent and the generation rate was 2 gal/min. No metals were present in the distillate product above 1 mg/kg and the water content was approximately 4 percent.

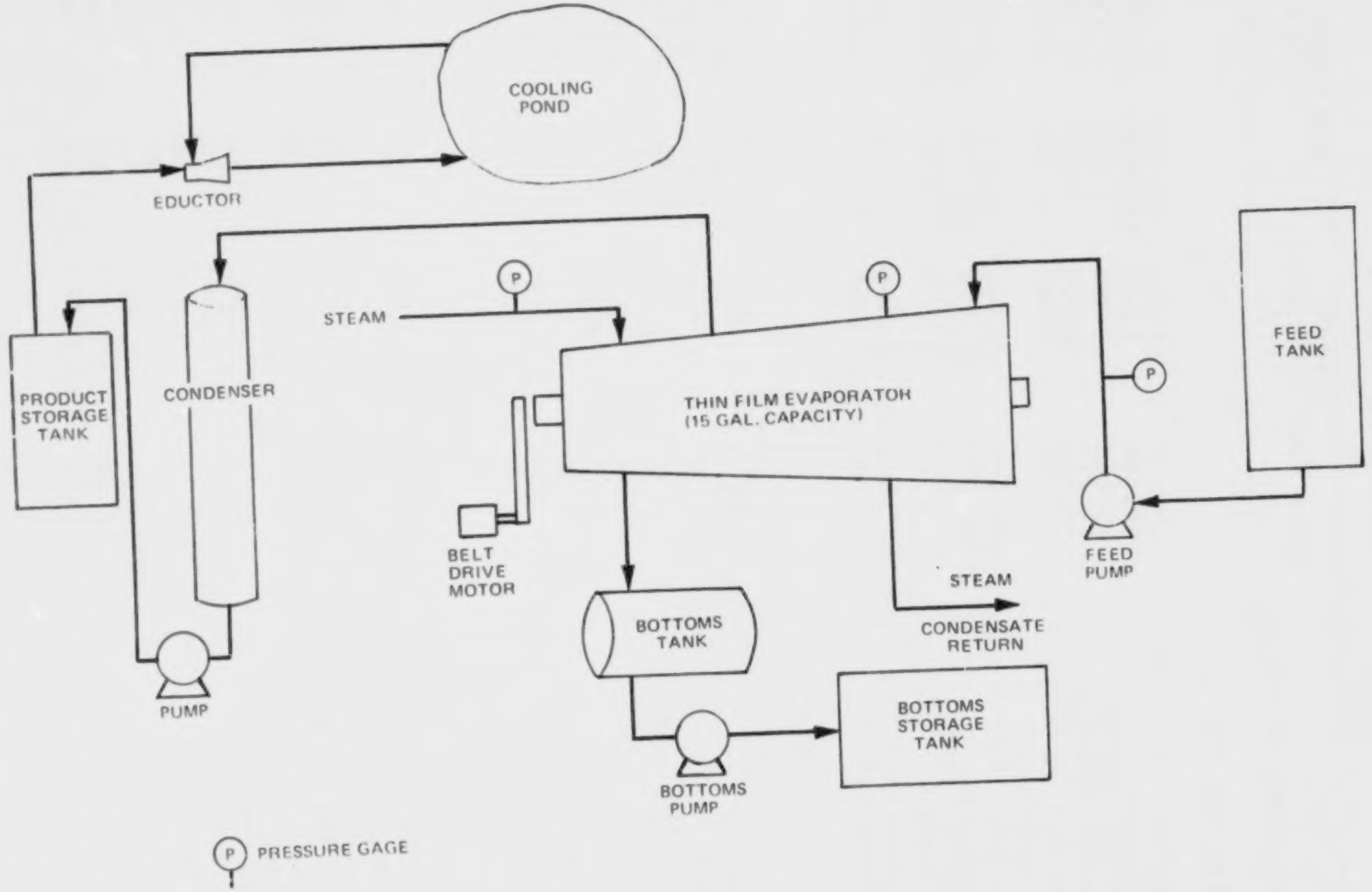
COOLING POND
EDUCTOR
PRODUCT STORAGE TANK
CONDENSER
PUMP
STEAM
THIN FILM EVAPORATOR (15 GAL. CAPACITY)
BELT DRIVE MOTOR
BOTTOMS TANK
BOTTOMS PUMP
BOTTOMS STORAGE TANK
STEAM
CONDENSATE RETURN
FEED TANK
FEED PUMP
P
P PRESSURE GAGE

Figure 10. Thin film evaporator process schematic - Facility L.

TABLE 24. FACILITY L - THIN FILM EVAPORATOR ANALYTICAL RESULTS

Analytical Parameter	Waste Feed (1)	Still Bottoms (2)	Distilled Product (3)		
			A	B	C
Volatiles (mg/kg)					
Xylene (m-,o-,p-)	66,000	210,000	80,000	87,000	86,000
Acetone	190,000	5,200	190,000	180,000	180,000
Ethyl Acetone	11,000	<12,000	12,000	12,000	11,000
Ethyl Benzene	22,000	48,000	27,000	29,000	29,000
Methyl Isobutyl Ketone	14,000	5,600	16,000	16,000	16,000
n-Butyl Alcohol	11,000	<5,500	13,000	13,000	13,000
Toluene	220,000	81,000	240,000	240,000	250,000
Methyl Ethyl Ketone	180,000	57,000	220,000	220,000	230,000
Isobutanol	3,800	<5,500	<3,000	<3,000	7,600
Isopropanol	76,000	<5,500	82,000	81,000	88,000
Metals (mg/kg)					
Antimony	0.87	4.0	0.015	<0.014	<0.012
Barium	*3.0	*17	*0.005	*0.005	*0.005
Cadmium	1.20	8.4	0.002	0.002	0.002
Chromium	9.5	81	0.014	0.011	0.012
Copper	<0.25	<0.50	0.07	0.10	0.07
Lead	*11	*100	*0.04	*0.04	*0.04
Mercury	<0.13	0.58	0.22	0.33	3.37
Nickel	0.90	4.2	0.002	0.004	0.003
Titanium	193	1,100	0.26	0.23	0.23
Zinc	14	100	0.39	0.49	0.48
Fuel					
Heating Value (Btu/lb)	14,698	14,795	NA	NA	NA
Viscosity (cSt)	2.825	26.156	NA	NA	NA
Density (g/cc)	0.8420	0.9370	0.83	0.83	0.83
Flash Point (deg C)	20	27	NA	NA	NA
Ash Content (%)	0.14	0.59	NA	NA	NA
Carbon (%)	70.62	72.76	NA	NA	NA
Hydrogen (%)	10.37	9.65	NA	NA	NA
Nitrogen (%)	0.91	1.14	NA	NA	NA
Oxygen (%)	14.29	6.51	NA	NA	NA
Chlorine (%)	0.24	0.17	NA	NA	NA
Sulfur (%)	0.49	0.42	NA	NA	NA

TABLE 24 (Continued). FACILITY L - THIN FILM EVAPORATOR ANALYTICAL RESULTS

Analytical Parameter	Waste Feed (1)	Still Bottoms (2)	Distilled Product (3) A	B	C
Miscellaneous					
Water Content (%)	2.94	8.76	4.02	2.98	4.44
Total Chloride (%)	0.18	0.20	0.15	0.13	0.15

NA - Not Analyzed. *Value not supported by QC data.

Residue Characterization --

The still bottoms stream was two-phased, the top portion (80-90 percent by volume) being brown and the bottom phase being gray and semisolid. The major organic constituent found to be present was xylene (210,000 mg/kg). Metal concentrations in the still bottoms stream were more concentrated than in the feed, as would be expected. Titanium was present at 1,100 mg/kg. Results of the analysis of the TFE still bottoms, according to the TCLP tests, are shown in Table 25. All of the metals were below the regulatory levels published in the Federal Register (CFR, Vol. 51, No. 114, 6/13/86) but toluene and methyl ethyl ketone were above the specified levels.

The heat content of the still bottoms stream is slightly higher than the waste feed and is significant at 14,800 Btu/lb. The water content of the still bottoms is 8.76 percent.

Evaluation of Effectiveness

The thin film evaporator at Facility L is effective at recovering nonhalogenated solvents and in producing still bottoms that can be utilized as supplemental fuel in offsite asphalt kilns. A yield (recovery) of 54 percent of total solvents from the feed material was achieved.

In spite of the higher water content of the still bottoms stream compared with the feed stream, the bottoms heat content (14,800 Btu/lb) is slightly higher than the feed stream heat content. With this fuel value the bottoms are suitable for use as a supplemental fuel in offsite asphalt kilns.

Metal concentrations in the still bottoms stream are shown to be concentrated by factors of 6 to 9. Despite the high metals concentration the TCLP data shows that no metals exceed regulatory levels in the extract. The effect of these metals present during burning of the material as a supplemental fuel poses concerns regarding potential metal emissions. Though the metals met regulatory requirements for the TCLP compounds, such as methyl ethyl ketone and toluene, exceeded regulatory requirements. The thin film evaporator does allow volatile constituents to be present in the still bottoms to enhance its heat value and thereby promotes its use as an offsite supplemental fuel.

Residue Management

Still bottoms generated from the treatment of nonhalogenated waste streams are shipped offsite to be used as supplemental fuel in asphalt kilns.

TABLE 25. FACILITY L - TCLP DATA FOR THE STILL BOTTOMS DISCHARGED FROM THE TFE

Parameter	Concentration
Volatile Organics	
Acetone	260 mg/l
Ethyl Acetate	110 mg/l
Methyl Isobutyl Ketone	32 mg/l
n-Butyl Alcohol	140 mg/l
Toluene	41 mg/l
Methyl Ethyl Ketone	1,800 mg/l
Metals	
Barium	0.24 mg/l
Cadmium	0.13 mg/l
Chromium	0.33 mg/l
Lead	1.2 mg/l
Nickel	0.077 mg/l
Zinc	3.7 mg/l

SECTION 8

INCINERATION

Incineration can be used for destruction of organic hazardous wastes of any physical form - gas, liquid, slurry, sludge, or solid. Two incineration processes (one at Facility F and the other at Facility H) were included in the sampling program.

THEORY

Incineration is a controlled high temperature oxidation process by which hazardous organic wastes are destroyed. the combustion reactions of incineration convert organic compounds to gaseous carbon dioxide and water vapor. If the waste contains chlorinated organics, HCl and free chlorine are also generated as incineration by-products. Incineration of wastes containing organic sulfur or nitrogen would result in formation of SO_x and NO_x, respectively.

All incineration systems used to burn hazardous waste consist of a waste feed system, an air or oxygen-fed burner system, a combustion chamber, combustion monitoring systems and, if required, equipment for air pollution control and ash removal. Two types of incinerators commonly used for destruction of organic hazardous wastes are (1) liquid injection and (2) rotary kiln.

(1) Liquid Injection. The liquid injection system is capable of incinerating a wide range of gases and liquids. The combustion system has a very simple design with virtually no moving parts. A burner or nozzle atomizes the liquid waste and injects it into the combustion chamber where it burns in the presence of air or oxygen. A forced draft system supplies the combustion chamber with air to provide oxygen for combustion and turbulence for mixing. The combustion chamber is usually a cylinder lined with refractory (i.e., heat resistant) brick.

(2) Rotary Kiln. Rotary kiln systems are capable of incinerating solid, liquid, and gaseous hazardous wastes either separately or simultaneously. In general, rotary kiln operators utilize high Btu liquid wastes in conjunction with lower Btu solids in order to enhance combustion. Because of their versatility, rotary kilns have been used for treatment in large commercial facilities. A rotary kiln is a slowly rotating, refractory-lined cylinder that is mounted at a slight incline from the horizontal. Solid wastes enter at the high end of the kiln, and liquid or gaseous wastes enter through atomizing nozzles in the kiln or afterburner section. Rotation of the kiln exposes the solids to the heat, vaporizes them, and allows them to combust by mixing with air. The rotation then causes the ash to move to the lower end of the kiln where it can be removed. Rotary kiln systems usually have a secondary combustion chamber or afterburner following the kiln to ensure more complete combustion of the volatilized components of solid wastes.

Important operating variables for either type of incinerator are temperature, residence time, turbulence or mixing and the ratio of oxygen to combustible material.

PROCESS DESCRIPTIONS

Facility F

Facility F is a commercial hazardous waste treatment facility accepting drummed organic hazardous wastes from a variety of industrial generators. Typical wastes accepted for incineration at Facility F include chlorinated and nonchlorinated solvent wastes (F001, F002, F003, F004, F005), paint formulating wastes, furniture strippers, printing inks and dyes, polymer wastes, agricultural products, pharmaceutical production wastes, lab packs and contaminated soils. The facility does not accept Polychlorinatedbiphenyls (PCBs), dioxins, compressed gases, radioactive waste, or organic wastes containing significant concentrations of potassium, sodium, lithium, mercury or lead.

Figure 11 shows a process flow diagram for Facility F. Waste organic liquids are pumped from drums into one of four 6000 gallon steel blend tanks. Waste liquids are selected such that the final blend has greater than 8000 BTU/lb and less than 40% chlorine.

Drums containing primarily solids, nonpumpable materials or residues remaining from liquid pumping are transported to the drum processing building. Operators use an air-driven cutting tool to remove the tops of steel drums. Drums are then upended and dumped onto a coarse-mesh screen above a steel tank. Any free liquids drain into the tank and are subsequently pumped to a blend tank. Solids remaining on the screen are shoveled into 30-gallon fiber packs. Empty drums are steam-cleaned, and the cleaning solutions are added to a blend tank.

The incinerator at Facility F is a dual chamber unit with a thermal input rating of 19.8 million BTU/hr. All wastes are introduced into the lower chamber. Liquid waste from the blend tank is injected into this chamber by atomization with steam. Solids in fiber packs are loaded into the incinerator by a ram feeder. Solids residence time in the lower chamber is estimated at 15 to 30 minutes. The upper chamber is an afterburner used to complete combustion. Fuel oil is burned in this chamber to maintain required temperature.

Dry ash residue is conveyed from the lower chamber out of the incinerator building and is dumped into a roll-off container. The ash is transported off-site to a permitted hazardous waste landfill.

A caustic scrubber is employed for removing air pollutants, particularly HCl, from the stack gas. This scrubber permits burning wastes with up to 40 percent total chlorine content.

The approximate feed and effluent flowrates during the sampling period were as follows:

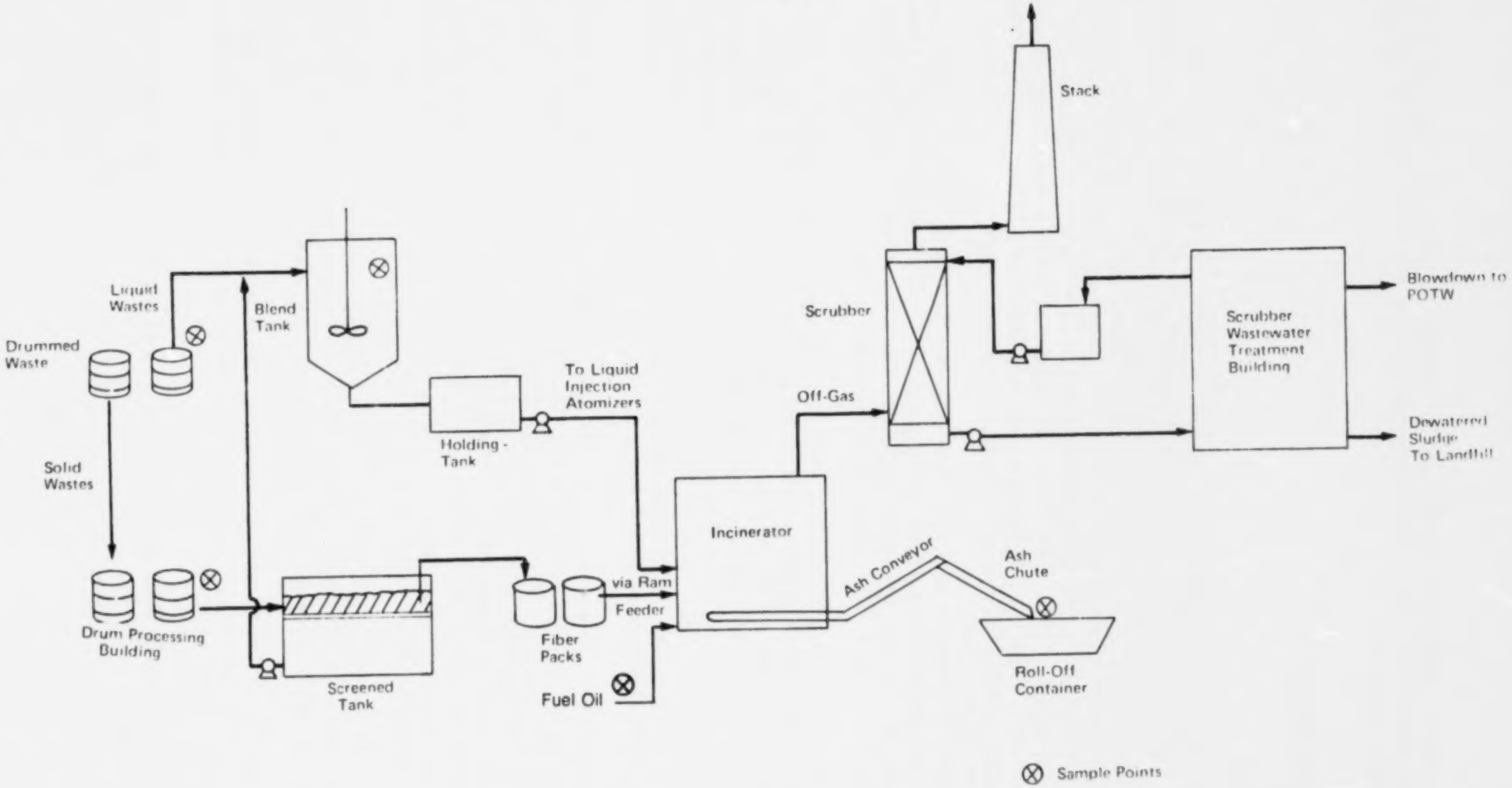
Drummed Waste
Liquid Wastes
Solid Wastes
Drum Processing Building
Blend Tank
Screened Tank
Holding - Tank
To Liquid Injection Atomizers
Fiber Packs
via Ram Feeder
Fuel Oil
Incinerator
Off-Gas
Ash Conveyor
Ash Chute
Roll-Off Container
Scrubber
Stack
Scrubber Wastewater Treatment Building
Blowdown to POTW
Dewatered Sludge To Landfill
⊗ Sample Points

Figure 11. Facility F process flow diagram.

Feed

Solid waste = 48.1 ft^3/hr (two 30-gallon fiber packs every 10 minutes

Liquid waste = 28.3 ft^3/hr (3.52 gallons per minute)

Residuals

Bottom Ash = 5.25 ft^3/hr (1 roll-off = 21 yd^3 filled in 36 hours)

Scrubber Wastewater = 722 ft^3/hr

Scrubber wastewater treatment includes pH adjustment, hydroxide and sulfide precipitation of heavy metals, clarification, pressure chamber filtration, and sludge dewatering with a recessed plate filter press (Figure 12).

Scrubber water collects in a 400 gallon steel tank and is pumped to the scrubber wastewater treatment building. The wastewater flow is typically 90 gpm.

The wastewater pH is adjusted to 9.0 with 25 percent caustic in a 7,000-gallon mixed tank. Sodium sulfide is added to precipitate heavy metal sulfides which remain in solution after hydroxide precipitation at pH 9.0. Ferric chloride is added to this tank as a conditioning agent to improve clarification and filtration. High cationic synthetic polyelectrolyte polymer is added before an in-line static mixer as the wastewater is pumped to the clarifier.

Wastewater is clarified in a 20,000 gallon steel tank which overflows to a second 20,000 gallon steel storage tank. Clarified effluent is then pumped through a seven element filter. The elements are operated in parallel and each element contains a 200 mesh polypropylene filter bag. The filter is backwashed once each shift, and the backwash water is returned to the clarifier.

A portion of the treated filtered wastewater is recycled to the scrubber. A blowdown stream is stored in a third 20,000 gallon steel tank. Blowdown wastewater from this storage tank is trucked periodically to the municipal wastewater treatment plant.

Sludge from the clarifier is pumped to a 7,000 gallon steel conditioning tank, Synthetic polyelectrolyte is added to this mixed tank as a filter aid. The conditioned sludge is pumped to a recessed plate filter press once each shift. The filtrate is returned to the clarifier, and the dewatered sludge is collected for landfill disposal. During the sampling episode the dewatered sludge passed the EP Toxicity test for metals and was considered non-hazardous.

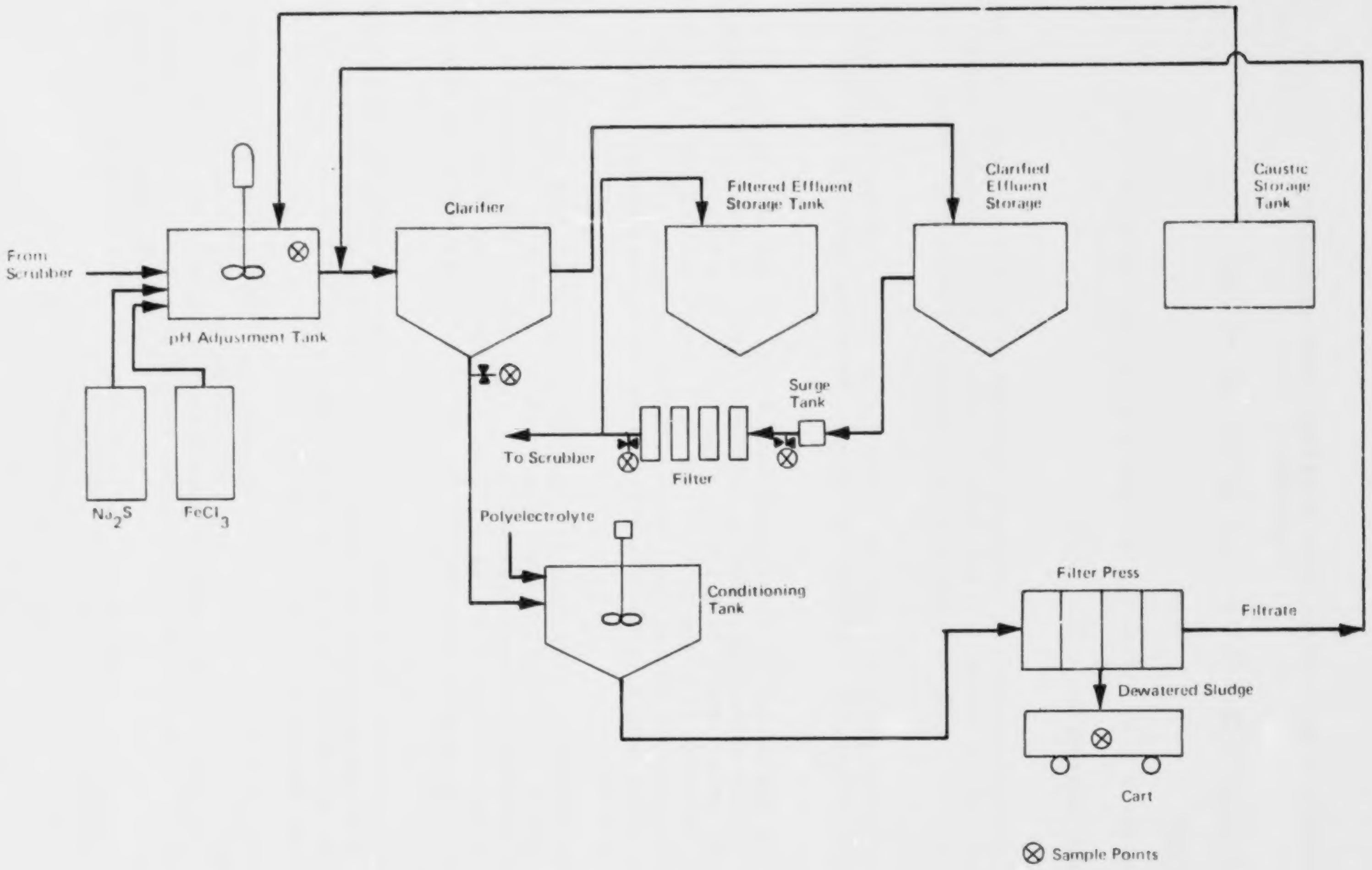
From Scrubber
pH Adjustment Tank
Na_2S
$FeCl_3$
Clarifier
Filtered Effluent Storage Tank
Clarified Effluent Storage
Caustic Storage Tank
Surge Tank
To Scrubber
Filter
Polyelectrolyte
Conditioning Tank
Filter Press
Filtrate
Dewatered Sludge
Cart
⊗ Sample Points

Figure 12. Facility F scrubber wastewater treatment.

Facility H

Facility H is a chemical manufacturing plant owned and operated by a major chemical company. Both solid and liquid hazardous organic wastes generated at Facility H and at several of the company's other plants are incinerated at Facility H.

Solid wastes and sludges are received in drums and typically include paint sludges, chlorinated hydrocarbons, coke solids, vacuum filter solids, waste filter elements and polymeric tar.

Liquid wastes are transported by tanker truck and are transferred on site to one of four 20,000 gallon waste storage tanks. One is used as a feed tank, another as a waste blending tank, and the remaining two as holding tanks. Liquid wastes commonly include waste organic solutions, waste solvents, tank farm nitriles and chloroprene catalyst sludge.

The incineration system (Figure 13) at Facility H includes both a rotary kiln incinerator and a single-stage liquid injection incinerator. The total system thermal input rating is 40 million BTU/hr. Both the incinerators use natural gas as auxiliary fuel. The liquid injection incinerator burns only liquid wastes, which are predominantly generated from the manufacturing operations on site. Ash is removed about four times per year from the liquid injection incinerator. The rotary kiln incinerator will typically burn the same liquid wastes, in addition to a variety of solid wastes.

Solid wastes are all contained in drums. The drums are fed to the kiln by an automated feed system. First the drum top is removed, and the drum is lifted vertically up an elevator. It is then transported horizontally across a conveyor. The drum then enters an airlock before being inverted and the contents dumped into the rotary kiln. Empty drums are returned down the elevator and placed in storage. If the drums must be cleaned they are washed with toluene or water and set out to dry. The toluene and water wastes are also incinerated.

The rotary kiln is followed by a natural gas-fired afterburner designed to further burn gases exiting the kiln. Normal operating temperatures for the rotary kiln are 1200-1400°F. Afterburner temperatures for the rotary kiln are 100-150°F higher than the kiln. Average solid residence times in the kiln are between 40 and 60 minutes, and the kiln rotation is between 0.10 and 0.20 revolutions per minute. Ash is discharged continuously from the kiln to an ash sluice.

Liquid wastes, as described previously, are stored in four (20,000 gallon) tanks. These tanks are recirculated by pumps to mix the tank contents. It normally takes 2 to 3 days to burn the contents of one tank.

The liquid incinerator is air atomized and care must be taken to prevent plugging of the feed lines. Tanks and feed lines are flushed with toluene between burning incompatible liquid wastes. Normal operating temperatures for the liquid incinerator ranges from 1400 to 1600°F.

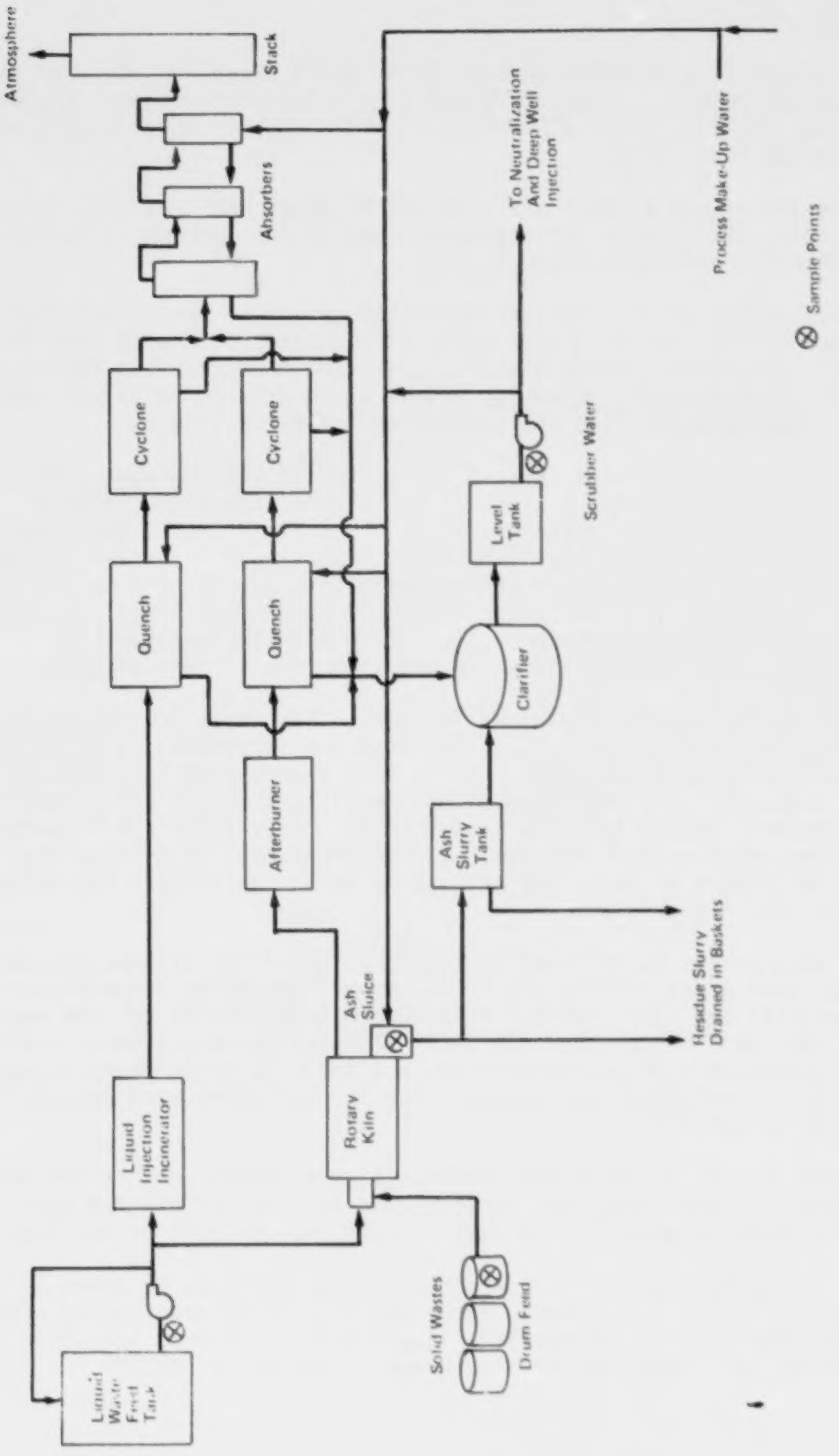
Atmosphere
Stack
Absorbers
To Neutralization And Deep Well Injection
Process Make-Up Water
Sample Points
Cyclone
Cyclone
Scrubber Water
Quench
Quench
Level Tank
Clarifier
Afterburner
Ash Slurry Tank
Ash Sluice
Residue Slurry Drained in Baskets
Liquid Injection Incinerator
Rotary Kiln
Solid Wastes
Drum Feed
Liquid Waste Feed Tank

Figure 13. Facility II process flow diagram.

Each of the two incinerators is followed by a water quench and a cyclone. The quench serves to cool effluent gases. the cyclone acts to remove entrained particulates and droplets prior to scrubbing the exhaust gases.

Gases exiting the two cyclones combine to form a single exhaust stream. the combined gas stream then passes through a three-stage packed tower absorption (scrubber) system for removal of particulates and HCl.

There are two waste streams generated by this incineration facility: scrubber wastewater and ash residue. A portion of the scrubber blowdown (about 10-12,000 gal/hr) is neutralized and processed at an on-site wastewater treatment facility prior to deep well injection. Ash residue is typically allowed to accumulate in the ash sluice and slurry tank for about four weeks. The rotary kiln is shut down approximately once a month for ash removal. Ash is disposed of in on-site RCRA permitted hazardous waste landfills.

SAMPLING PROGRAMS

Facility F

The on-site sampling program at Facility F took place during a six-day period in June 1986. Solid and liquid waste volume-composite samples were collected during the first three days of this period. Scrubber wastewater treatment background grab samples were collected shortly before incineration was started. Ash and scrubber wastewater treatment time-composite samples were collected during the incineration run, which lasted for thirty-six hours. Sample points are indicated on Figures 11 and 12. These locations and the sampling procedures used are described in Table 26.

Facility H

The on-site sampling program at Facility H took place during a six-day period from August 28 through September 2, 1986. Three discrete liquid waste grab samples were taken from the blend tank being fed to the incinerator. Three discrete grab samples of each of five solid waste types were collected from individual drums. Three scrubber wastewater grab samples were collected during the incineration run. After the incineration run was terminated, the incinerator was allowed to cool down for about 48 hours before six discrete ash grab samples were procured from various locations. Sample points are indicated on Figure 15. These locations and the sampling procedures used are described in Table 27.

TABLE 26. FACILITY F - SAMPLE LOCATIONS AND PROCEDURES

Sample	Description of Procedure	Type	Time Interval
Liquid Wastes	Samples were collected from individual drums using disposable glass COLIWASAS (or, in cases where drums contained primarily solids with a liquid layer, a stainless steel beaker was dipped into the open drum to obtain a liquid sample). Samples from individual drums making up a batch from a single generator were composited in a stainless steel bucket. Buckets were covered with aluminum foil and iced continuously during sample collection.	Composite	One sample obtained for each discrete waste influent
Solid Wastes	A stainless steel trowel was used to collect a solid sample from each drum or from the screen after each drum was dumped. Samples from individual drums making up a batch from a single generator were composited in a stainless steel bucket. Buckets were covered with aluminum foil and iced continuously during sample collection.	Composite	One sample obtained for each discrete solid waste influent
Blend Tank	Equal volume grab samples were taken from each of two sample taps on the side of the completely mixed blend tank and composited in a stainless steel bucket.	Composite	One sample for each blend tank after all liquid wastes had been added and mixed.
Fuel Tank	Sample was collected from the fuel oil storage tank using a disposable glass COLIWASA inserted through the tank filling port.	Grab	One sample prior to beginning of incineration run.

TABLE 26. (Continued) FACILITY F - SAMPLE LOCATIONS AND PROCEDURES

Sample	Description of Procedure	Type	Time Interval
Ash Residue	Grab samples of ash were collected by holding a stainless steel bucket beneath the ash chute. Individual grab aliquots were composited by mixing with a stainless steel trowel in a stainless steel bucket.	Composite	A grab aliquot was collected every two hours during the incineration run, beginning four hours after the first solid waste was fed to the incinerator.
pH Tank Background	Sample was collected from the mixed pH adjustment tank using a stainless steel beaker on a sample pole.	Grab	One sample prior to start of incineration run.
Clarified Effluent Background	Sample was collected from the sample tap immediately upstream of the Ronningen-Petter filter using a stainless steel beaker.	Grab	Same as above
Filtered Effluent Background	Sample was collected from the sample tap immediately downstream of the Ronningen-Petter filter using a stainless steel beaker.	Grab	Same as above
Treatment Residue-Background	Sample was collected from the clarifier underflow pipe at the open hose connection with the valve open. A stainless steel bucket was used.	Grab	Same as above
pH Tank Batch #1	Grab aliquots were collected from the mixed pH adjustment tank using a stainless steel beaker on a sample pole and composited in a stainless steel bucket. Individual VOC grab samples were collected in sealed vials every four hours.	Composite	A grab aliquot was collected every hour during the incineration run beginning, four hours after burning of liquid waste from the blend tank started.

TABLE 26. (Continued) FACILITY F - SAMPLE LOCATIONS AND PROCEDURES

Sample	Description of Procedure	Type	Time Interval
Clarified Effluent Batch #1	Grab aliquots were collected from the sample tap immediately upstream of the Ronningen-Petter filter using a stainless steel beaker and composited in a stainless steel bucket.	Composite	Same as above
Filtered Effluent Batch #1	Grab aliquots were collected from the sample tap immediately downstream of the Ronningen-Petter filter using a stainless steel beaker and composited in a stainless steel bucket. Individual VOC grab samples were collected in sealed vials every four hours.	Composite	Same as above
Treatment Residue-Batch #1	Grab aliquots were collected from the underflow pipe at the open hose connection with the valve open. A stainless steel bucket was used. Individual grab aliquots were composited in a stainless steel bucket.	Composite	Same as above
Dewatered Residue-Batch #1	A stainless steel trowel was used to collect samples from various locations in the wheeled cart after residue from the filter press was discharged to this cart. Sample was composited by mixing individual grabs with the trowel in a stainless steel bucket.	Composite	Grab aliquots were collected at the completion of the two filter press cycles occurring during the incineration run.

TABLE 27. FACILITY H - SAMPLE LOCATIONS AND PROCEDURES

Sample Description	Sample Location	Sampling Procedure
WASTE SAMPLES		
Liquid Waste Samples #1, #2, and #3	Discrete samples were collected from the sample tap on the recirculation/feed pump from blend tank #3.	After purging the sample line, waste was discharged from the feed tank to a 2 1/2 gallon capacity glass composite container, and then samples were fractioned into their respective sample containers.
Solid Waste Samples #1, #2, #3, #4, and #5	Discrete samples were collected directly from the drums containing the waste material Three discrete samples per waste lot were collected from the five major solid waste types incinerated. Only two solid waste samples were collected from lot 4 due insufficient waste volume.	Four drums for each of the five waste types burned were available for sampling. Discrete samples were collected from three drums of each waste type using stainless steel buckets and trowels.
Scrubber Water Samples #1, #2, and #3	Discrete samples were collected from the sample tap following the level tank.	After purging the sample line, the scubber water was discharged from the scrubber system to a 2 1/2 gallon capacity glass composite container, and then samples were fractioned into their respective sample containers.

TABLE 27. (Continued) FACILITY H - SAMPLE LOCATIONS AND PROCEDURES

Sample Description	Sample Location	Sampling Procedure
Ash Residue Samples #1, #2, #3, #4, #5 and #6	Six discrete samples of the ash residue were collected after the incinerator was allowed to cool down for about 48 hours. Four samples were collected from the top of the incinerator ash pile in the inclined manway where ash accumulated. The fifth sample was collected from the top of the accumulated ash in the slurry tank. The sixth sample was procurred from the outer layer of residue that accumulated on the kiln refractory.	Stainless steel trowels were used to sample the accumulated ash in the sluice manway and the slurry tank. The residue was placed directly into the sample containers with the trowels. The residue sample from the kiln refractory was collected by air hammering the outermost ash into a stainless steel bucket from a number of locations. The residue was then homogenized and placed into the respective sample containers.
BACKGROUND SAMPLES		
Field Blank	Deionized water sample was collected from the laboratory deionized water column as a field blank	Deionized water sample was carried through all sampling steps as a field/laboratory blank.
Industrial Water Supply	Discrete sample to document background levels of contamination in process make-up water was collected from a hose located adjacent to the waste treatment clarifier.	After purging the process water line and hose for about 5 minutes, sample containers were filled directly from the process water line.

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*QA/QC sample

DATA

Waste Characterization

Facility F --

During the week-long sampling program at Facility F, samples of five solid wastes and five liquid wastes were obtained. In addition, a sample of the liquid waste from the blend tank was taken. As Table 28 shows, the major volatile organic components of these wastes were the common solvent compounds acetone, benzene, chloroform, methylene chloride, methyl ethyl ketone, tetrachloroethylene, toluene, trichloroethylene and xylene. These nine components account for about 54 percent by weight of the liquid waste blend. Surprisingly, the liquid waste contained 28.7 weight percent water and its fuel value was only 6,824 BTU/lb, below the plant's stated goal of >8000 BTU/lb. The liquid blend's organic chloride content of 27.99 percent was below the plant's stated upper limit of 40 percent.

The five solid wastes represent diverse industrial sources. Information supplied to Facility F by the waste generators indicates that the five solid wastes were (1) epoxy resins, (2) used nylon mesh filter bags, (3) waste tetrachloroethylene and wax mixture, (4) waste methylene chloride and polymer, and (5) eye shadow with tetrachloroethylene.

Measurable levels of chromium, copper, lead, nickel, and zinc were found in all waste samples. The highest metal concentration was 759 mg/l copper in the liquid waste blend.

Facility H --

During a week-long sampling period, Facility H incinerated the contents of one 20,000 gallon liquid blend tank along with 183 drums representing five different types of solid waste. The solid waste types were: (1) chlorinated hydrocarbon coke from dichlorobutene synthesis, (2) polymeric tar, (3) filtered organo-metallic waste solids from adiponitrile area, (4) adiponitrile spent filter cartridges, and (5) scrap paint solids. Analytical data summarizing the chemical and physical characteristics of each waste are presented in Table 29.

Toluene is the major volatile organic component of the waste. Several semivolatile compounds were reported in one or more of the waste samples. Copper and nickel are the two metals present at highest concentrations in the liquid waste and they are reported at significant levels in each of the solid wastes as well. Solid waste #5, the scrap paint solids, exhibits the highest metals concentration with high levels of lead, chromium and zinc, three common pigment metals.

The liquid waste blend had a water content of about 17 percent, an organic halide content of 14.24 percent and a fuel value of 11, 926 BTU/lb. The fuel value of the solid wastes ranged from 5,866 BTU/lb for the polymeric tar to 12,000 BTU/lb for the scrap paint solids.

TABLE 28. FACILITY F WASTE CHARACTERIZATION

PARAMETER	Liquid Blend	Solid #1	Solid #2	Solid #3	Solid #4	Solid #5
		Epoxy Resins	Solvent Filter Bags	Waste Tetrachloroethylene and Wax	Waste Methylene Chloride & Polymer	Eye Shadow with Tetrachloroethylene
VOLATILE ORGANICS (ppb)						
Acetone	4,035,100	210,000	<23,000	<24,000	<24,000	<25,000
Benzene	20,000	<5,000	<12,000	<12,000	<12,000	<12,000
Chloroform	29,300	<5,000	<12,000	<12,000	<12,000	<12,000
Methylene Chloride	46,413,200	<5,000	<12,000	<12,000	34,000,000	<12,000
Methyl Ethyl Ketone	2,937,900	<10,000	<23,000	<24,000	<24,000	<25,000
Tetrachloroethylene	385,904,000	<5,000	<12,000	260,000,000	580,000	220,000,000
Toluene	140,366,500	32,350,000	8,900,000	100,000	<56,000	24,000
Trichloroethylene	1,372,600	<5,000	<12,000	8,600,000	<12,000	<12,000
Xylene	828,000	9,242,500	<12,000	<12,000	620,000	<12,000
TOXIC METALS (ppm)						
Antimony	<1.0	<1.0	<1.0	<1.0	<1.0	4.0
Arsenic	0.19	<0.1	0.8	0.1	<0.1	0.1
Barium	111	41.0	70.0	<1.0	<1.0	125
Beryllium	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Cadmium	<0.5	<0.5	1.3	<0.5	0.7	1.2
Chromium-hexavalent	1.83	<0.01	0.540	0.097	0.350	0.300
Chromium-total	20.3	12.0	61.0	12.0	0.8	40.0
Copper	759	13.0	43.0	11.0	2.0	75.0
Lead	34.6	20.0	110.0	2.0	6.0	5.0
Mercury	0.16	<0.1	1.4	<0.1	0.2	0.3
Nickel	8.7	12.0	70.0	45.0	0.8	6.0

TABLE 28 (Continued). FACILITY F WASTE CHARACTERIZATION

PARAMETER	Liquid Blend	Solid #1 Epoxy Resins	Solid #2 Solvent Filter Bags	Solid #3 Waste Tetrachloroethylene and Wax	Solid #4 Waste Methylene Chloride & Polymer	Solid #5 Eye Shadow with Tetrachloroethylene
Selenium (ppm)	1.6	7.0	7.0	6.0	3.0	<1.0
Silver	<0.2	<0.2	0.4	<0.2	<0.2	<0.2
Thallium	<1.0	<1.0	3.0	3.0	<1.0	2.0
Zinc	61.2	8.0	105.0	19.0	7.0	1100.0
OTHER ANALYSES						
WT % Carbon	32.25	68.60	4.89	80.95	13.70	9.64
WT % Hydrogen	7.02	9.08	3.40	9.51	9.44	7.07
WT % Water	28.7	0.49	32.88	1.15	11.25	1.38
WT % Organic Halide	27.99	0.31	0.57	52.41	24.47	22.91
Sp. Gravity	1.084	1.0215	1.3745	1.3135	1.1105	1.5954
Total Solids (mg/kg)	156,000	745,100	581,200	250,800	428,600	662,800
BTU/lb.	6,824	15,850	DNI	5,750	7,019	4,021

DNI = Does Not Ignite

TABLE 29. FACILITY H WASTE CHARACTERIZATION*

PARAMETER	Liquid	Solid #1	Solid #2	Solid #3	Solid #4	Solid #5
	Blend	Chlorinated Hydrocarbon Coke	Polymeric Tar	Filtered Organo-Metallic Waste Solids	Spent Filter Cartridges	Scrap Paint Solids
VOLATILE ORGANICS (ppb)						
Carbon Disulfide	37,000	<62,000	<62,000	<62,000	ND	<62,000
Benzene	47,300	<62,000	<62,000	<62,000	52,000	<62,000
Toluene	340,000,000	10,200,000	<62,000	4,900,000	850	11,700,000
Ethylbenzene	80,300	<62,000	<62,000	72,000	ND	4,600,000
Styrene	181,700	<62,000	<62,000	59,000	ND	<62,000
Xylenes	47,900	<62,000	<62,000	340,000	ND	20,200
4-methyl - 2-pentanone	<10,000	<120,000	<120,000	<120,000	ND	250,000
SEMIVOLATILE ORGANICS (ppb)						
Benzyl Alcohol	1,300,000	<7,500	<60,000	<190,000	ND	<7,500
1,2 dichlorobenzene	1,867,000	<7,500	<60,000	357,000	ND	<7,500
n-nitrosodiphenylamine	3,233,000	<7,500	<60,000	497,000	ND	<7,500
naphthalene	<50,000	<7,500	<60,000	490,000	ND	60,300
2-methylnaphthalene	<50,000	<7,500	<60,000	2,900,000	ND	<7,500
acenaphthene	<50,000	<7,500	<60,000	70,700	ND	<7,500
phenol	<50,000	<7,500	<60,000	<190,000	184,000	<7,500
di-n-butyl phthalate	<50,000	<7,500	<60,000	<190,000	ND	22,300
butyl benzyl phthalate	<50,000	<7,500	<60,000	<190,000	ND	217,000
TOXIC METALS (ppm)						
Antimony	<1	<1	4	116	<1	12
Arsenic	<0.1	<0.1	<0.1	0.21	<0.1	<0.1
Barium	<1	<1	<1	21	4.5	29
Beryllium	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Cadmium	<0.5	<0.5	<0.5	1.3	<0.5	<0.5
Chromium-hexavalent	0.23	.09	0.26	SI	0.015	0.085

TABLE 29 (Continued). FACILITY H WASTE CHARACTERIZATION*

PARAMETER	Liquid Blend	Solid #1 Chlorinated Hydrocarbon Coke	Solid #2 Polymeric Tar	Solid #3 Filtered Organo-Metallic Waste Solids	Solid #4 Spent Filter Cartridges	Solid #5 Scrap Paint Solids
Chromium - total (ppm)	2.2	2.1	0.83	67	10	992
Copper	68	5,700	85	74	1,013	347
Lead	1.7	<1	<1	30	<1	3,470
Mercury	<0.1	<0.1	<0.1	0.9	0.4	<0.1
Nickel	227	91	1.2	500	1,200	39
Selenium	<1	<1	<1	<1	<1	<1
Silver	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Thallium	<1	<1	<1	<1	<1	<1
Zinc	4.8	1.8	1.0	195	60.5	1,670
OTHER ANALYSES						
Wt% Carbon	54.96	48.00	43.12	29.09	IS	50.44
Wt% Hydrogen	8.46	3.61	6.70	6.69	IS	6.45
Wt% Water	17.16	33.91	7.45	27.61	IS	0.52
Wt% Organic Halide	14.24	---	---	---	---	---
Wt% Silica	<0.01	0.77	0.44	15.35	6.99	2.81
Sp. Gravity	0.9576	1.0548	1.2325	1.1313	1.0431	1.1191
Total Solids (mg/kg)	73,000	565,600	659,800	626,033	132,550	604,200
BTU/lb.	11,926	6,243	7,944	5,866	IS	12,000

SI=Sample Interference
IS=Insufficient Sample
* Each value represents the average of three grab samples.

Residue Characterization

Facility F --

The wastes described in the previous section were incinerated during a 36-hour period. Beginning 4 hours after the first fiberpack of solid waste was fed to the incinerator, grab samples of ash were collected from the ash chute every two hours until the incineration run was complete. The individual grab samples were composited to form a single ash residue sample representative of the burn. Tables 30 and 31 summarize the analytical data for this sample.

Compositional analysis of the ash shows significant concentrations of several volatile and semivolatile organic compounds. The toxicity characteristic leaching procedure was run on the ash sample. Extract levels of two volatile organics - methylene chloride and tetrachloroethylene - exceed the regulatory levels for these compounds, implying that the ash must be classified as a hazardous waste.

Metals appear to have concentrated in the ash as expected, with most metals present in the ash at levels greater than their concentrations in any of the waste samples. Arsenic exhibits the greatest concentration factor. The composition of arsenic in the ash - 42 mg/kg - is 52 times the highest waste sample arsenic concentration. Copper at 13,800 mg/kg is the major metal component of the ash. Both TCLP and EP extracts were analyzed for toxic metals. None of the eight listed EP Toxicity metals were found above regulatory levels in either extract. Comparison of extract and compositional metals levels indicates that the ash is fairly resistant to leaching of metals.

The ash had a total solids content of about 60 percent by weight and a specific gravity of 1.28.

Data on the scrubber wastewater and its treatment products are summarized in Table 32. With the exception of acetone which was also found in the laboratory blank, none of the organic compounds measured in the wastes or ash were detected in the scrubber wastewater or the filtered effluent.

Seven toxic metals were reported above detection levels in the scrubber wastewater at concentrations ranging from 0.6 mg/l for arsenic to 11.0 mg/l for copper. Metal concentrations in the filtered effluent are less than those of the scrubber wastewater.

No information is available on Facility F's discharge permit to the local publicly owned treatment works (POTW). However, strictly for purposes of comparison, Table 33 presents filtered effluent data for five metals along with the daily maximum concentrations of metals permitted to be discharged to a POTW by metal finishing operations (Effluent Guidelines for Metal Finishing. Amended September, 1984). Facility F's effluent exceeds the metal finishing effluent guidelines concentrations for copper, lead and zinc.

TABLE 30. FACILITY F ASH ANALYTICAL DATA* - ORGANICS

PARAMETER	Composition (μg/kg)	TCLP Extract (μg/L)
Volatile Organics		
Methylene chloride	38,000	8,800
Acetone	20,000	<3,300
Chloroform	46	<1,700
2-Butanone	2,000	<3,300
1,1,1 trichloroethane	12	<1,700
1,2 dichloroethane	32	<1,700
trichloroethylene	120	<1,700
benzene	42	<1,700
4-methyl-2-pentanone	2,300	<3,300
tetrachloroethylene	1,200,000	48,000
toluene	2,500	11,000
chlorobenzene	27	<1,700
ethylbenzene	380	<1,700
styrene	320	<1,700
xylenes	1,900	<1,700
methanol	410,000	N.M.
Semivolatile Organics		
Phenol	40,000	<1,400
Nitrobenzene	29,000	<200
2,4 dimethyl phenol	23,000	<1,000
naphthalene	24,000	310
2-nitroaniline	180,000	1,300
dimethyl phthalate	55,000	370
diethyl phthalate	120,000	410
di-n-butyl phthalate	160,000	<200

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[*] Analysis of a single composite sample. Aliquots of ash making up the composite were collected every two hours during the incineration run.

TABLE 31. FACILITY F ASH ANALYTICAL DATA* - METALS

PARAMETER	Compositional (mg/kg)	TCLP Extract (mg/L)	EP Toxicity Extract (mg/L)	EP Toxicity Standards (mg/L)
Toxic Metals				
Antimony	8.0	0.094	<1.0	None
Arsenic	42.0	0.062	0.2	5.0
Barium	150	0.026	<1.0	100.0
Beryllium	<0.2	<0.005	<0.2	None
Cadmium	2.0	0.02	<0.5	1.0
Hexavalent Chromium	0.083	NM	NM	None
Total Chromium	71.0	0.01	<0.3	5.0
Copper	13,800	0.729	4.0	None
Lead	30.0	<0.05	<1.0	5.0
Mercury	0.2	0.00025	<0.1	0.2
Nickel	190	1.14	2.0	None
Selenium	<1.0	<0.001	<1.0	1.0
Silver	0.4	<0.005	<0.2	5.0
Thallium	2.0	<0.001	<1.0	None
Zinc	180	1.15	0.3	None
Other Analyses				
Total solids (mg/kg)	599,300	-	-	-
Specific gravity (g/mL)	1.2809	-	-	-
Paint Fitler Test	PASS	-	-	-

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[*] Analysis of a single composite sample. Aliquots of ash making up the composite were collected every two hours during the incineration run.

TABLE 32. FACILITY F SCRUBBER WASTEWATER TREATMENT DATA

PARAMETER	Scrubber Wastewater (pH Tank)	Filtered Effluent	Dewatered Sludge	Dewatered Sludge EP Toxicity Extract
Volatile Organics (μg/l)				
Methylene Chloride	<5.0	<5.0	390 B	-
Acetone	32 B	28 B	170 B	-
Chloroform	<5.0	<5.0	<18	-
Methyl ethyl ketone	<10.0	<10.0	<36	-
1,1,1 trichloroethane	<5.0	<5.0	<18	-
1,2 dichloropropane	<5.0	<5.0	<18	-
trichloroethylene	<5.0	<5.0	<18	-
benzene	<5.0	<5.0	<18	-
4-methyl-2-pentanone	<10.0	<10.0	<36	-
tetrachloroethylene	<5.0	<5.0	<18	-
toluene	<5.0	<5.0	<18	-
chlorobenzene	<5.0	<5.0	<18	-
ethyl benzene	<5.0	<5.0	<18	-
styrene	<5.0	<5.0	<18	-
xylenes	<5.0	<5.0	<18	-
methanol	NM	NM	NM	-
Semivolatile Organics (μg/L)				
Phenol	<20.0	<20.0	<740	-
Nitrobenzene	<20.0	<20.0	<740	-
2,4 dimethyl phenol	<20.0	<20.0	<740	-
naphthalene	<20.0	<20.0	3,600	-
2-nitroaniline	<100	<100	<3,700	-
dimethyl phthalate	<20.0	<20.0	<740	-
diethyl phthalate	<20.0	<20.0	<740	-
di-n-butyl phthalate	<20.0	<20.0	<740	-

TABLE 32 (Continued). FACILITY F SCRUBBER WASTEWATER TREATMENT DATA

PARAMETER	Scrubber Wastewater (pH Tank)	Filtered Effluent	Dewatered Sludge	Dewatered Sludge EP Toxicity Extract
Toxic Metals (mg/L)				
Antimony	4.0	<1.0	313	<1.0
Arsenic	0.6	0.5	140	0.2
Barium	<1.0	<1.0	183	<1.0
Beryllium	<0.2	<0.2	<0.2	<0.2
Cadmium	0.7	<0.5	400	<0.5
Hexavalent Chromium	0.526	0.063	<0.01	NM
Total Chromium	1.4	<0.3	55	<0.3
Copper	11.0	5.0	8300	1.0
Lead	2.0	1.3	1600	<1.0
Mercury	<0.1	<0.1	60	<0.1
Nickel	4.0	1.7	2200	<0.3
Selenium	<1.0	<1.0	<1.0	<1.0
Silver	<0.2	<0.2	0.6	<0.2
Thallium	<1.0	<1.0	3.0	<1.0
Zinc	8.0	5.0	3100	0.5
Other Analyses				
Total Solids (mg/L)	86,600	65,700	450,400	-
Total Dissolved Solids (mg/L)	82,500	65,400	NM	-
Total Suspended Solids (mg/L)	670	360	NM	-
Specific Gravity	1.0540	1.0373	1.2921	-
pH	>12	>12	NM	-
Alkalinity (mg/L as $CaCO_3$)	NM	NM	37,888	-

NM = not measured

B = Analyte found in blank as well as in sample. Value shown has not been corrected for blank contamination.

TABLE 33. FACILITY F EFFLUENT METALS

Metal	Filtered Effluent (mg/L)	Metal Finishing Effluent Guidelines (mg/L)
Copper	5.0	3.38
Nickel	1.7	3.98
Lead	1.3	0.69
Chromium	<0.3	2.77
Zinc	5.0	2.61

The dewatered sludge from scrubber wastewater treatment was about 45 percent total solids by weight. Metals concentrations in the sludge were greater than 1000 ppm for copper, lead, nickel and zinc. An EP Toxicity test was done on this sludge. None of the metals exceeded regulatory levels in the extract.

Facility H --

Analytical data for Facility H ash is summarized in Table 34. No volatile or semivolatile organic compounds were found in the ash from Facility H. This ash had a total solids content of about 81 weight percent and a specific gravity of about 1.94. The ash contained nearly 18 weight percent silica. Measurable concentrations of eight toxic metals were reported, with copper (4,600 mg/kg), nickel (4,200 mg/kg) and zinc (1,160 mg/kg) being present at the highest concentrations. Although none of the regulated TCLP metals were found above regulatory level in the TCLP extract, extract levels of copper, nickel and zinc were quite high.

Facility H's scrubber wastewater is very acidic, with a pH of less than 1.0. Four toxic metals - copper, lead, nickel and zinc - are present at concentrations greater than 25 mg/l. No volatile or semivolatile organic compounds were found in the scrubber wastewater. Table 35 presents the analytical data for this wastewater stream.

EVALUATION OF EFFECTIVENESS

The effectiveness of an incinerator and its associated air pollution system is normally measured in terms of destruction and removal efficiency (DRE), as defined by the following formula:

$$DRE = \frac{W_{in} - W_{out}}{W_{in}} \times 100\%$$

W_{in} = mass feed rate of the principal hazardous constituent(s) to the incinerator

W_{out} = mass emission rate of the principal organic hazardous constituent(s) to the atmosphere as measured in the stack prior to discharge.

Both incineration systems had demonstrated >99.99% DRE for principal organic hazardous constituents during trial burns required for RCRA permits. No stack sampling was done during this program to verify trial burn performance. Assuming, however, that > 99.99% DRE is routinely achieved, then both incineration systems are very effective in destroying hazardous organic wastes.

The DRE formula considers only off-gas emissions to the atmosphere, ignoring the other residues of incineration - ash, scrubber wastewater, and

TABLE 34. FACILITY H ASH ANALYTICAL DATA* - METALS

PARAMETER	Compositional (mg/kg)	TCLP Extract (mg/l)	EP Toxicity Standards (mg/l)
TOXIC METALS			
Antimony	14.5	0.03	None
Arsenic	<0.1	0.004	5.0
Barium	75	0.39	100.0
Beryllium	<0.2	0.002	None
Cadmium	<0.5	0.01	1.0
Hexavalent Chromium	0.05	<10	None
Total Chromium	361	0.085	5.0
Copper	4,600	96	None
Lead	340	0.56	5.0
Mercury	1.6	0.004	0.2
Nickel	4,200	34	None
Selenium	<1	<0.05	1.0
Silver	<0.2	<0.005	5.0
Thallium	<1	<0.001	None
Zine	1,160	25	None
OTHER ANALYSES			
Silica (WT%)	17.77	-	-
Total Solids (mg/kg)	811,000	-	-
Specific Gravity	1.9363	-	-

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* Each value represents the average of six grab samples.

TABLE 35. FACILITY H SCRUBBER WASTEWATER DATA*

PARAMETER	Scrubber Wastewater
Toxic Metals (mg/kg)	
Antimony	<1
Arsenic	<0.1
Barium	2
Beryllium	<0.2
Cadmium	<0.5
Chromium-hex	0.2
Chromium-total	1.5
Copper	241
Lead	106
Mercury	<0.1
Nickel	27
Selenium	<1
Silver	<0.2
Thallium	<1
Zinc	363
Other Analyses	
Total Solids (mg/kg)	3867
Total Dissolved Solids (mg/kg)	3500
Total Suspended Solids (mg/kg)	67
Total Organic Halide (wt%)	0.08
Total Chlorine (wt%)	1.59
Specific Gravity (g/mL)	0.9936
pH	0.7

*Each value represents the average of 3 grab samples.

scrubber wastewater treatment sludge. This sampling program focused in the collection and characterization of these solid and liquid residues.

Properly designed and operated incineration systems should achieve virtually complete destruction of organic compounds. This appears to be the case at Facility H, where no detectable levels of volatile or semivolatile organics were found in either the ash or scrubber wastewater. Facility F's ash did contain significant concentrations of organic compounds. This may be indicative of incomplete combustion due to inadequate retention time, mixing, or temperature in the incinerator. A rough mass balance shows that about 0.06 percent of the major solvent component of the Facility F liquid waste was found in the ash residue. In other words, comparison of the waste fed to the incinerator and the corresponding ash indicates greater than 99.93 percent destruction. Table 36 summarizes the mass balance. Data from both facilities indicate that significant levels of toxic metals are present in waste solvents and organic process wastes. Since metals are not destroyed by incineration, they tend to concentrate in the treatment residues - ash and scrubber wastewater.

RESIDUE MANAGEMENT

Incineration of organic hazardous waste results in the formation of ash. The ash residue from both facilities F and H were disposed in RCRA permitted hazardous waste landfills.

Air pollution control devices used to remove particulates and inorganic pollutants (HCl, NO_x, SO_x) from incinerator off-gases also generate treatment residues. Facility F uses a caustic scrubber. Scrubber wastewater is treated by chemical precipitation, coagulation, sedimentation and filtration to remove particulates and dissolved metals. Most of the treated water is recycled to the scrubber. A blowdown stream is collected in an on-site storage tank and periodically trucked to the local POTW. Treatment of the scrubber wastewater results in a dewatered metal sulfide sludge (filter cake) which is shipped off-site for disposal in a RCRA-permitted hazardous waste landfill.

Facility H uses a water scrubber. The resulting acidic scrubber wastewater is pumped to an on-site deep injection well for disposal.

TABLE 36. APPROXIMATE MASS BALANCE - FACILITY F

Stream	Flowrate (L/hr)	Specific Gravity	Tetrachloro-ethylene Concentration	Tetrachloro-ethylene Mass Flow Rate (kg/hr)	% of Total Feed TCE
Liquid Waste Feed	801.2	1.084	385,904 mg/l	309.20	84.20
Solid Waste #1	127.2	1.0215	<5,000 mg/kg	-	-
Solid Waste #2	133.0	1.3745	<12,000 mg/kg	-	-
Solid Waste #3	115.7	1.3135	260,000 mg/kg	39.50	10.80
Solid Waste #4	63.6	1.1105	580 mg/kg	0.04	.01
Solid Waste #5	52.1	1.5954	220,000 mg/kg	18.30	4.99
Total Feed	1292.8			367.04	100.00
Ash	148.6	1.2809	1,200 mg/kg	0.228	0.062

SECTION 9

WASTE BLENDING/FUEL SUBSTITUTION

THEORY

Fuel substitution involves the use of combustible organic wastes as substitutes for conventional fuels that are burned in high temperature industrial processes such as industrial boilers and kilns. As in incineration, the organic waste is destroyed in flame combustion yielding essentially carbon dioxide and water. If the waste contains chlorinated organics, hydrogen chloride and chlorine are also produced. In most cases, the waste is not the sole fuel, but is used to supplement primary fuels such as natural gas, fuel oil, or coal. The result is that the total fuel cost is reduced because the fuel value of the waste is utilized concurrent with its high temperature destruction.

In order to be used as a fuel substitute, an organic waste must have certain physical and chemical characteristics within acceptable ranges. The most important characteristics are heating value, viscosity, "pumpability", solids content, moisture content, chlorine concentration, and the presence of potential air pollutant constituents. Reaction kinetics is also an important factor when considering the organic waste and type of incineration process to be used (i.e., matching of time-temperature profiles for the waste and incineration process).

The heating value of a waste, expressed as BTU/lb, is the heat released when the waste is burned. A minimum heating value of about 8000 BTU/lb is required to sustain combustion of a liquid waste. If an organic waste exhibits a low heating value, this is usually reflective of a high moisture content or a high concentration of halogenated compounds. An upper limit of about 5 or 6 percent organic chlorine for a fuel substitute is usually imposed in order to maintain sufficient heating value for sustained combustion and to limit the concentration of HCl and free chlorine in the combustion gas.

Pumpability is generally required to allow handling of the fuel substitute in a system designed to burn fuel oil. The Kinematic viscosity is an important parameter affecting the degree of atomization achievable in any burner.

The presence of metals and specific hazardous constituents (e.g. PCB's) in a waste fuel substitute may necessitate special air emission controls and special treatment of the resulting ash.

Wastes that are unacceptable for fuel substitution due to low heating value, high chlorine concentration, high ash content, or high viscosity may be blended with higher quality organic wastes or with fuel oil to produce a mixture of acceptable quality for fuel substitution. The blending process may include grinding or shredding of solid organic wastes to allow their incorporation into pumpable liquid fuel substitute mixtures.

PROCESS DESCRIPTION

Facility B's organic waste blending operation is illustrated in Figure 14. Organic liquid wastes are received in drums which often contain a bottom layer of sludge and/or solids. To incorporate organic solids and sludges into the fuels, Facility B uses a mechanical process to grind solids such as resins, latex, still bottoms and paint sludges to a uniform particle size of less than 800 microns. Liquid and settled solids are pumped from the drums to a vacuum truck then through a grinder. Following the grinder, the blend passes through a coarse grinder pump, a trash pump, a filter and a fine grinder pump before being transferred to a blend/storage tank. Suspension of the ground solids in the liquid is maintained by agitation of the fuel substitute mixture in blend/storage tanks both at Facility B and at the receiving facility. Any residual material which cannot be pumped out of the drums is solidified with lime and disposed of in a hazardous waste landfill.

Currently, 8,000 to 12,000 gallons of organic waste can be processed daily. Future plans are to increase the fuel blending capacity to about 20,000 gallons per day. Construction of five new tanks for incorporation as part of the fuel blending program is nearly complete.

SAMPLING PROGRAM

Sampling of Facility B's waste to fuel blend process took place during a two-day period in July 1985. One batch of low chlorine content waste was processed each day. For each batch, samples of the organic waste influent and the treated fuel blend after grinding were procured. In addition, two composite samples of nonpumpable organic sludge residue from the bottom of waste drums were procured each day. Each residue sample represents a composite of material from four drums.

The sample points are indicated in Figure 14. Exact sampling locations and sampling procedures are described in Table 37.

DATA

Waste Characterization

Two distinct batches of low chlorine fuel substitutes were blended during the sampling program. Table 38 summarizes the RCRA waste codes, waste descriptions and observations for these two batches.

Data for all organic waste blending operation samples are summarized in Table 39. During the preparation of batch 1, 3,000 gallons of low chlorine content waste was pumped from drums into the vacuum tank truck. A sample-designated "organic waste influent" - was taken from the truck at this point. Subsequently, 1,000 gallons of bottom waters from drums was added to the truck. Another sample - designated "combined waste influent" - was then taken from the truck. The batch 1 organic waste influent sample had a very low organic halide content, 0.074%. Its total solids content was

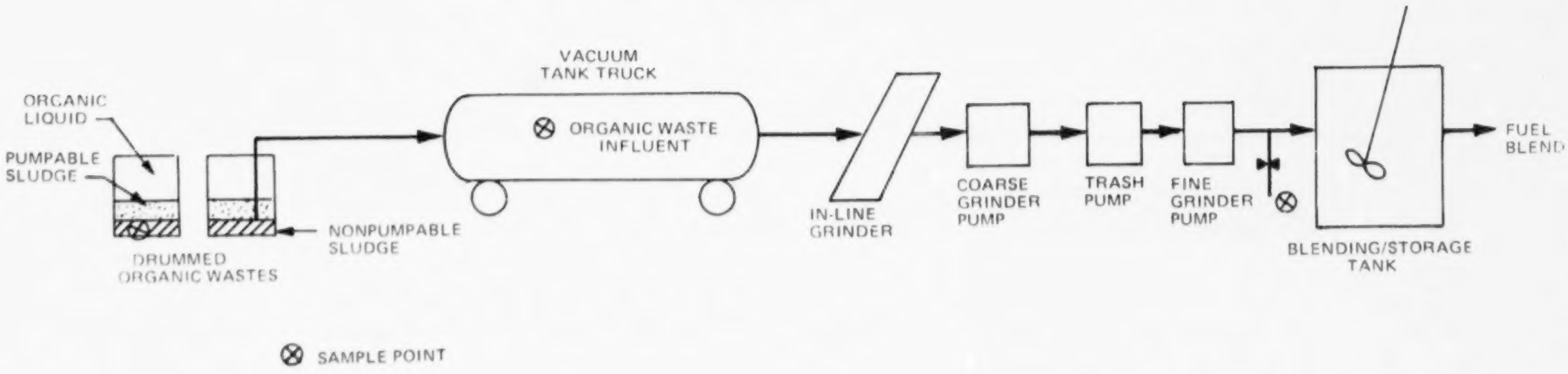
VACUUM
TANK TRUCK
ORGANIC
LIQUID
PUMPABLE
SLUDGE
ORGANIC WASTE
INFLUENT
NONPUMPABLE
SLUDGE
DRUMMED
ORGANIC WASTES
IN-LINE
GRINDER
COARSE
GRINDER
PUMP
TRASH
PUMP
FINE
GRINDER
PUMP
BLENDING/STORAGE
TANK
FUEL
BLEND
SAMPLE POINT

Figure 14. Flow diagram/sample points for organic waste blending at Facility B.

TABLE 37. FACILITY B SAMPLING LOCATIONS AND PROCEDURES

Sample Reference	Description	Type of Sample	Frequency
Organic Waste Influent	Sample was taken from tank truck using a glass COLIWASA.	Volume Composite	One sample per batch, taken after all organic waste to be blended has been pumped into the vacuum tank truck.
Combined Waste Influent	Sample was taken from tank truck using a glass COLIWASA.	Volume Composite	For batch 1 only. One sample taken after bottom water waste was added to organic waste in vacuum tank truck.
Treated Fuel Blend	A sample of the homogenized waste was collected from a sample siphon immediately downstream of the fine grinder pump.	Grab	One sample per batch.
Non Pumpable Sludge	Samples were procured from individual drums using a stainless steel trowel and composited in a stainless steel bucket.	Volume Composite	Two samples per batch. Each sample represents a composite of material from five drums.

TABLE 38. ORGANIC WASTE INCORPORATED INTO FUEL BLENDS

Batch	RCRA Waste Code(s)	Waste Description	Observations
1	D001	Paints and paint sludges	5 distinct layers - various suspended and settleable solids.
2	D001 F003	Varnish, paints, alcohols, xylene	3 distinct layers - suspended and settleable solids.

TABLE 39. FACILITY B FUEL BLENDING PROCESS DATA

		BATCH 1					BATCH 2			
Parameter	Units	Organic Waste Influent	Combined Waste Influent	Treated Fuel Blend	Nonpumpable Sludge #1	Nonpumpable Sludge #2	Organic Waste Influent	Treated Fuel Blend	Nonpumpable Sludge #3	Nonpumpable Sludge #4
BTU Content	BTU/lb	2,472	786	2,179	5,217	15,350	NM	11,601	8,443	14,765
% carbon	wt%	NM	NM	23.13	40.45	59.15	NM	58.17	40.75	64.85
% hydrogen	wt%	NM	NM	10.17	7.94	10.33	NM	10.15	6.94	10.91
% water	wt%	NM	NM	80.56	NM	NM	NM	11.14	NM	NM
% organic halide	wt%	0.074	0.26	0.78	NM	NM	2.32	0.79	NM	NM
Total solids	ppm	69,000	130,000	120,000	410,000	560,000	380,000	390,000	530,000	690,000
Total volatile solids	ppm	58,000	110,000	100,000	240,000	540,000	310,000	350,000	260,000	620,000
Total suspended solids	ppm	26,000	46,000	70,000	NM	NM	NM	330,000	NM	NM
Total dissolved solids	ppm	43,000	84,000	50,000	NM	NM	NM	60,000	NM	NM
Total volatile suspended solids	ppm	21,000	37,000	55,000	NM	NM	NM	280,000	NM	NM
Total volatile dissolved solids	ppm	37,000	73,000	45,000	NM	NM	NM	70,000	NM	NM
Viscosity	cst	NM	NM	<21	NM	NM	NM	<21	NM	NM
Metals										
Cadmium	mg/kg	NM	NM	NM	220	3,400	NM	NM	5,000	30,000
Chromium (total)	mg/kg	NM	NM	NM	390	260	NM	NM	170	1,400
Copper	mg/kg	NM	NM	NM	3,900	5,100	NM	NM	510	800
Lead	mg/kg	NM	NM	NM	36,000	70,000	NM	NM	8,600	5,900
Nickel	mg/kg	NM	NM	NM	62	260	NM	NM	31	650
Zinc	mg/kg	NM	NM	NM	1,300	8,500	NM	NM	610	14,000
EP Toxicity										
Arsenic	mg/L	NM	NM	NM	<0.10	<0.10	NM	NM	<0.10	<0.10
Barium	mg/L	NM	NM	NM	<1.0	<0.10	NM	NM	<0.10	<0.10
Cadmium	mg/L	NM	NM	NM	17.0	0.06	NM	NM	5.0	0.05
Chromium	mg/L	NM	NM	NM	13.0	<0.05	NM	NM	0.10	<0.05
Lead	mg/L	NM	NM	NM	255	13.0	NM	NM	<0.10	0.56
Mercury	mg/L	NM	NM	NM	<0.10	<0.10	NM	NM	<0.10	<0.10
Selenium	mg/L	NM	NM	NM	6.0	<1.0	NM	NM	<1.0	<1.0
Silver	mg/L	NM	NM	NM	<0.20	<0.20	NM	NM	<0.20	<0.20

69,000 ppm, of which 84 percent was volatile at 550°F. The heating value was measured at 2,472 BTU/lb. The total solids concentration for batch 1 combined waste influent sample (75 percent low chlorine material and 25 percent bottom waters) during period two was 130,000 ppm, of which 85 percent is volatile at 550°F.

In comparison to other samples collected, the heating value of this sample was very low (786 BTU/lb), presumably the result of the high water content of the sample. In contrast, the batch 2 organic liquid waste sample had a total solids content of 390,000 ppm, with a volatile solids content at 550°F of 350,000 ppm. The heating value of the waste was much higher (11,614 BTU/lb) than the batch 1 waste samples, undoubtedly due to the lower water content (only 11.14 percent by weight). Organic waste influent viscosities could not be determined due to particulate clogging of the Zahn cups. A waste sample from each batch was manually mixed, placed in an Imhoff cone, and allowed to settle for an hour. The batch 1 sample was observed to stratify into five distinct vertical zones. Similarly, the batch 2 unblended influent waste sample was observed to settle into three layers ranging from a thin, dark colored oil at the top to an extremely coarse, dense layer at the bottom of the Imhoff cone.

Product Characterization

As a result of the grinding process, the solids composition of the raw organic waste samples was observed to change in three areas. First, the suspended solids concentration increased for both treated samples collected. Second, the large particles that had hindered measurement of the raw waste viscosity were broken apart and kinematic viscosity measurements of less than 21 centistokes (CST) were obtained for the both batch 1 and batch 2 samples. Third, the settleable solids observations in the Imhoff cone were changed dramatically - no longer were distinct stratified layers present; instead a homogenous material with a thin oil band at the top of the Imhoff cone was observed. These sample profiles did not change during the next 48 hours, even in the absence of agitation. Figure 15 illustrates the results of the settleability test.

Residue Characterization

Two composite samples of nonpumpable organic waste material corresponding to each batch were collected from drums after pumping. These residues were analyzed for metals, solids, EP Toxicity, TCLP, % carbon, % hydrogen, and BTU content. Significant concentrations of six toxic metals - cadmium, chromium, copper, lead, nickel and zinc - were reported in the samples. Lead concentrations ranged from 5,900 ppm to 70,000 ppm. Cadmium concentrations ranged from 220 ppm to 30,000 ppm. The heating value of the samples also exhibited a wide variation, ranging from 5,217 BTU/lb to 15,350 BTU/lb. In contrast, the total solids concentration averaged 548,000 ppm with a relative standard deviation of 21 percent. The ratio of the percent weight of carbon to percent weight of hydrogen was about six to one for each of the four samples.

TCLP analysis was performed on a composite made up from the four nonpumpable organic sludge samples. Results are summarized in Table 40. No

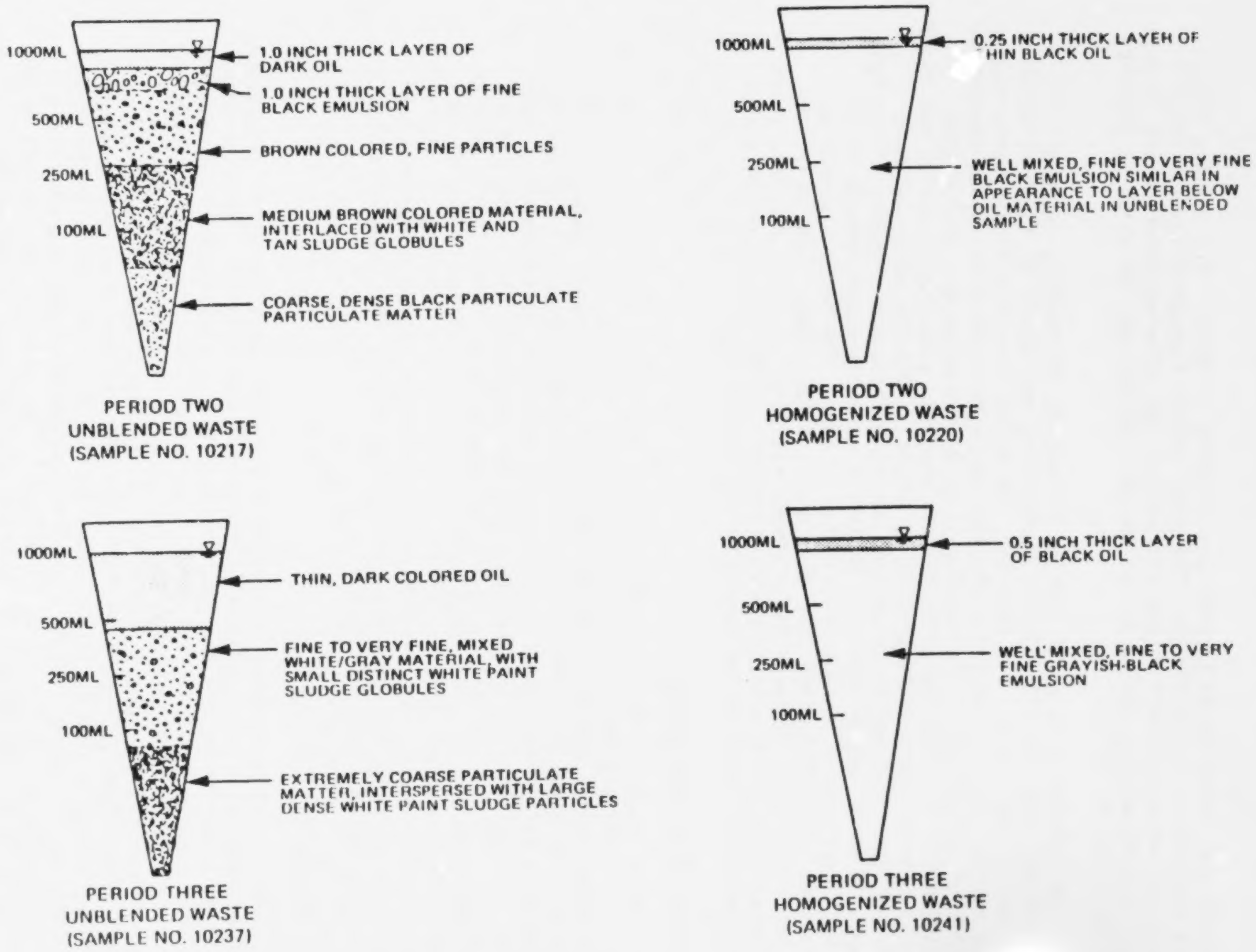
1000ML
500ML
250ML
100ML
1.0 INCH THICK LAYER OF DARK OIL
1.0 INCH THICK LAYER OF FINE BLACK EMULSION
BROWN COLORED, FINE PARTICLES
MEDIUM BROWN COLORED MATERIAL, INTERLACED WITH WHITE AND TAN SLUDGE GLOBULES
COARSE, DENSE BLACK PARTICULATE PARTICULATE MATTER
PERIOD TWO UNBLENDED WASTE (SAMPLE NO. 10217)
0.25 INCH THICK LAYER OF THIN BLACK OIL
WELL MIXED, FINE TO VERY FINE BLACK EMULSION SIMILAR IN APPEARANCE TO LAYER BELOW OIL MATERIAL IN UNBLENDED SAMPLE
PERIOD TWO HOMOGENIZED WASTE (SAMPLE NO. 10220)
THIN, DARK COLORED OIL
FINE TO VERY FINE, MIXED WHITE/GRAY MATERIAL, WITH SMALL DISTINCT WHITE PAINT SLUDGE GLOBULES
EXTREMELY COARSE PARTICULATE MATTER, INTERSPERSED WITH LARGE DENSE WHITE PAINT SLUDGE PARTICLES
PERIOD THREE UNBLENDED WASTE (SAMPLE NO. 10237)
0.5 INCH THICK LAYER OF BLACK OIL
WELL MIXED, FINE TO VERY FINE GRAYISH-BLACK EMULSION
PERIOD THREE HOMOGENIZED WASTE (SAMPLE NO. 10241)

Figure 15. Settleability analysis for the organic waste blending program at Facility B

TABLE 40. FACILITY B NON PUMPABLE SLUDGE TCLP DATA

Pollutant	Actual Compositional Analysis (mg/kg)*	TCLP Extract (mg/L)	EPA TCLP Reg. Level (mg/L)
VOLATILE ORGANIC COMPOUNDS			
Chloromethane	<100	<25	-
Bromomethane	<100	<25	-
Vinyl Chloride	<100	<25	0.05
Chloroethane	<100	<25	-
Methylene Chloride	320	<25	8.6
Acetone	2400	<250	-
Carbon Disulfide	<40	<10	14.4
1,1-dichloroethene	<40	<10	-
1,1-dichloroethane	<40	<10	-
Trans-1,2-dichloroethene	<40	<10	-
Chloroform	<40	<10	0.07
1,2-dichloroethane	<40	<10	0.4
Methyl ethyl ketone	9200	690	7.2
1,1,1-trichloroethane	340	<10	30
Carbon tetrachloride	<40	<10	0.07
Vinyl acetate	<40	<10	-
Bromodichloromethane	<40	<10	-
1,2-dichloropropane	<40	<10	-
Trans-1,3-dichloropropene	<40	<10	-
Trichloroethene	<40	<10	-
Dibromochloromethane	<40	<10	-
1,1,2-trichloroethane	<40	<10	-
Benzene	<40	<10	1.2
cis-1,3-dichloropropene	<40	<10	0.07
2-chloroethylvinylether	<40	<10	-
Bromoform	<40	<10	-
2-hexanone	1000	<50	-
4-methyl-2-pentanone	6700	210	-
Tetrachloroethene	74	<10	-
1,1,2,2-tetrachloroethane	<40	<10	1.3
Toluene	78000	150	14.4

TABLE 40 (Continued). FACILITY B NON PUMPABLE SLUDGE TCLP DATA

Pollutant	Actual Compositional Analysis (mg/kg)*	TCLP Extract (mg/L)	EPA TCLP Reg. Level (mg/L)
Chlorobenzene	<40	<10	1.4
Ethylbenzene	930	1.9	-
Styrene	<40	<10	-
Total Xylenes	4300	12	-
Acrylonitrile	NP	ND	-
Isobutanol	NP	ND	-
Pyridine	NP	ND	-
1,1,1,2-tetrachloroethane	NP	ND	-
METALS			
Arsenic	NP	<0.020	5
Barium	NP	0.32	100
Cadmium	NP	3.2	1
Chromium	NP	0.1	5
Lead	NP	1.5	5
Mercury	NP	0.0008	0.2
Selenium	NP	<0.040	1
Silver	NP	0.01	5

* = COMPOSITED AND ANALYZED SIX MONTHS AFTER SAMPLE COLLECTION

** = UNITS ARE NG/G

NP = COMPOSITIONAL ANALYSIS NOT PERFORMED

ND = NOT DETECTED

samples were procured after stabilization due to the limited processing schedule during the sampling episode. The actual compositional analysis of volatile organics indicates that, at the time of analysis (approximately six months after sample collection), several compounds (acetone, methyl ethyl ketone, 2-hexanone, 4-methyl-2-pentanone, toluene and total xylenes) were present in the sample at high concentrations. With respect to the proposed TCLP regulatory levels, the nonpumpable organic sludge composite sample exceeds the proposed criteria for methyl ethyl ketone and toluene.

Due to the long holding time and subsequent loss of volatile organics in the TCLP samples, the results are not valid for any regulatory support or other data use. TCLP results were obtained for purposes of increasing familiarity with a new testing protocol.

EVALUATION OF EFFECTIVENESS

The objective of the organic waste blending program is to segregate incoming waste, according to chlorine and moisture content, grind the waste material to facilitate materials handling and atomization of the waste, and blend the waste materials to provide a final product with a market outlet. Barometers to evaluate the effectiveness of this process include measurement of fuel value, chlorine content and moisture content, and observations regarding the viscosity and homogeneity of the material before and after grinding.

The heating value of the batch 1 fuel blend, which had a water content of 80.56 percent, was only 2,179 BTU/lb - well below the 8000 BTU/lb minimum requirement for a fuel product. This problem could be corrected by blending this material with higher quality organic wastes. The batch 2 fuel blend had an acceptable fuel value of 11,601 BTU/lb and a water content of 11.14 percent. The total organic halide content of both fuel blends was well below the 5 percent upper limit for low chlorine fuel blends.

As previously mentioned, both viscosity and homogeneity were clearly changed as a result of the grinding process. The large grit particulates that clogged the Zahn cups during the viscosity measurements of the raw organic waste samples were successfully shredded to less than 800 microns, thus generating treated fuel blends with kinematic viscosities of less than 21 cSt. The change in the homogeneity of the material as a result of the grinding operation was even more dramatic. The raw waste samples were a combination of paint sludges, varnishes, tar-like sludges, and gelatinous material interspersed with oils, solvents and water. Upon placing this material into Imhoff cones, the separation of the waste into the layers shown in Figure 15 transpired in about 15 minutes. The treated material (subjected to grinding) was observed to be extremely uniform in appearance. At the end of 48 hours of observation, the material showed no signs of settling into discrete zones, other than a thin layer of oil at the top of each sample. In summary, the waste segregation, blending and grinding operation appears to be very successful at completing the physical property objectives of the waste blending program.

RESIDUE MANAGEMENT

Nonpumpable sludges and solids which cannot be incorporated into fuel blends at Facility B are solidified with lime prior to land disposal.

The ultimate residues from the waste blending/fuel substitution process are ash, emissions to the atmosphere and any air pollution control residues (e.g. scrubber wastewater) which are generated during the combustion of the fuel. These ultimate residues were not available for sampling at Facility B, since the fuel blends are used off-site. Low chlorine fuel blends are sold to operators of kilns or furnaces that are permitted to store and burn EPA-listed hazardous wastes. The high chlorine waste blends, containing up to 45 percent chlorine, are sold to a manufacturer of low alkali cement for use in the cement kilns.

SECTION 10

STEAM STRIPPING

Three facilities using steam stripping to treat dilute aqueous waste streams are discussed in this section. The information on facility G was obtained from the sampling episode and post sampling report prepared by Metcalf & Eddy, Inc. Facilities M and N were sampled by Research Triangle Institute (RTI), and information reported herein was obtained from the following two reports prepared by RTI.

Facility M - "Field Test and Evaluation of the Steam Stripping Process at Plant H" (December 1986)

Facility N - "Field Test and Evaluation of the Steam Stripping Process at Plant I" (March 1987)

Table 41 is a summary of pertinent process parameters and waste stream data for the steam strippers at facilities G, M and N.

THEORY

Steam stripping is a continuous fractional distillation process in which live steam injected directly into the bottom of a packed or tray tower is used to strip volatile organic compounds from aqueous waste in multiple equilibrium stages. Figure 16 illustrates a typical steam stripping process. The liquid feed stream, preheated by heat exchange with the stripper bottoms, is introduced at the top of the column and flows countercurrent to the live steam. As the rising vapor contacts falling liquid at each stage, the more volatile components are transferred from the liquid to the vapor phase. The use of packed media or trays in the column allows for an increased liquid vapor transfer surface area to increase removal efficiencies. The overhead vapor (mixed steam and organic vapor) leaving the column is condensed in a water-cooled heat exchanger and collected in a product receiver or accumulator vessel. In most cases, where the organic contaminant(s) is not miscible with water, the condensed liquid will separate by gravity into two phases. The solvent phase may be recovered for reuse as solvent or fuel substitute or may be incinerated. The aqueous phase, saturated with the organic contaminant(s), is usually returned to the top of the column (reflux). Reflux enhances component preparation, increases the concentration of the stripped organics in the vapor stream, and is useful if the organics are to be recovered.

The bottoms or stripped effluent stream from a well designed and operated steam stripper is considerably reduced in organic content and may be discharged to a municipal sewer or an NPDES outfall. Carbon adsorption can be used to further reduce levels of volatile and semi-volatile compounds in the stripper effluent prior to discharge. Parameters which can be used to predict the flexibility of steam stripping specific organic compounds are boiling point and Henry's Law constants. Compounds with boiling points

TABLE 41. PROCESS PARAMETERS AND WASTE STREAM PROFILES FOR FACILITIES G, M AND N STEAM STRIPPERS

Facility	Type of Steam Stripper	Influent Flow Rate (l/min)	Bottoms Flow Rate (l/min)	Overhead Organic (l/min)	Condensate Aqueous (l/min)	Steam Flow Rate (kg/hr)	Feed/Steam Rate	Pretreatment or Post Treatment	Major Components in Waste Stream	Total VOC Influent (ppmw)	Total VOC Effluent (ppmw)	Percent Removal (%)
G	Packed, diameter = 8 in. Tower contains 10 ft of 5/8-in. pall rings.	19.7	21.8	0.283	-	120	10.5	None	Methylene chloride Chloroform	3,654	<5.66	>99.8
M	Tray (additional information is CBI)	816	845	3.2	20.3	1,700	28.8	None	1,2-Dichloroethane Chloroform	5,960	<9.8	>99.8
N	Packed with 1-in. saddles	40.7	44.7	0.14	1.9	253	9.6	Two (75,000 liter) solids decanters to pretreat stripper influent. Two granular activated carbon columns which are only used to treat the stripper effluent when it does not meet NPDES permit limitations.	Methylene chloride Chloroform	5,860	<0.037	>99.999

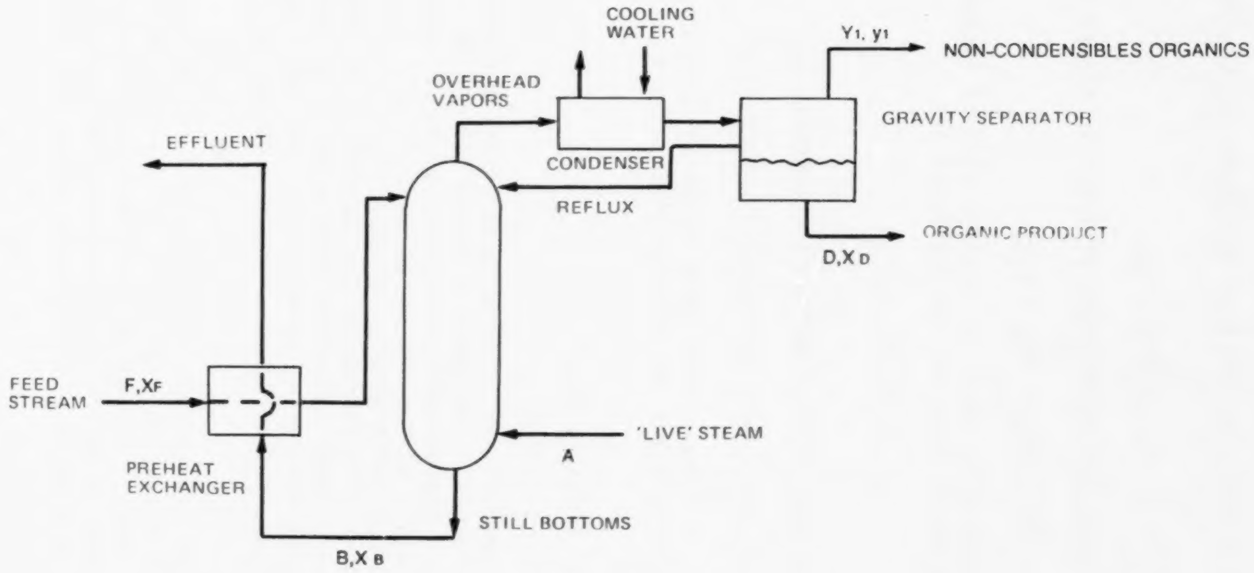
COOLING WATER
Y_1, y_1
NON-CONDENSIBLES ORGANICS
OVERHEAD VAPORS
GRAVITY SEPARATOR
EFFLUENT
CONDENSER
REFLUX
ORGANIC PRODUCT
D,X_D
FEED STREAM
F,X_F
'LIVE' STEAM
A
PREHEAT EXCHANGER
STILL BOTTOMS
B,X_B

Figure 16. Steam stripper schematic.

below 150°C or Henry's Law constants above 10^{-3} atm-m^3/mole are considered highly strippable.

A flow balance around the stripper in Figure 16 results in the equation:

$$F + A = B + D + V_1$$

The component mass balance for the stripper in Figure 16 is:

$$Fx_F = Bx_B + Dx_D + V_1y_1$$

Under equilibrium conditions the distribution of contaminant between the vapor phase and water phase can be expressed as:

$$y_i = (H_i/P)\ x_i$$

where:

y_i = Vapor phase mole fraction of component i

x_i = Liquid phase mole fraction of component i

H_i = Henry's law constant (atm) which is dependent on both system temperature and the concentration of the substance in solution

P = Total pressure (atm)

The steam stripper is most effective in treating aqueous solutions containing volatile organic compounds with low boiling points and which are immiscible in water. Removal of low solubility, higher boiling point compounds from non-volatile wastes can be achieved if the compounds form low boiling point azeotropes with water. Steam stripping is also useful in treating waste streams with compounds that decompose at their atmospheric boiling points, or for compounds which would present an ignition or explosion hazard if distilled using direct heat. Miscible organics such as ketones or alcohols are not stripped as readily using the steam stripping process.

Waste stream characteristics such as high concentrations of suspended solids, metals, and grease can also limit the use of steam stripping to remove volatile organics. Various pretreatment steps used to reduce these parameters include precipitation, coagulation, flocculation, centrifugation, membrane separation processes, flotation and other chemical/physical separation processes.

FACILITY G

Process Description

Facility G is a chemical manufacturing plant which produces a number of chemical intermediates that are ultimately used by the chemical, cosmetics and agricultural industries. The facility operates several unit processes including a methylene chloride steam stripping process.

The methylene chloride steam stripper treats wastes generated from on-site unit processes. The process waste to the steam stripper consists of methylene chloride, water, salt and organic residue (containing approximately 0.4 percent methylene chloride and 99.6 percent water).

Process waste feed is collected in two plastic-lined storage tanks. These tanks are referenced as VD-12 (10,000 gallons) and VD-15 (5000 gallons) in Figure 17. Both of these tanks have level measurement devices that are displayed in the operator control room. The feed is pumped from tanks VD-12 and VD-15 through a preheat exchanger and enters near the top of the steam stripper at an average flow rate of approximately 5 gallons per minute. A Foxboro magnetic flow tube is used to measure the process influent flow rate, with display in the control room. A rotameter has also been installed in-line as a backup flow measurement device. The steam stripping tower contains 10 feet of 5/8-inch pall rings and has a diameter of 8 inches. The column has approximately five theoretical stages based on pilot plant data. The column mid-point temperature and overhead temperature are measured by in-line temperature thermocouples. During the sampling episode the stripper was operated at approximately 101°C.

The treated or "stripped" effluent exits the bottom of the column and is collected in a 2,000-gallon agitated and baffled pressure reactor vessel (tank RD-02), prior to flowing by gravity to the preheat exchanger (used to preheat the incoming waste) and ultimately to the local publicly-owned treatment works (POTW). Pressure reactor vessel RD-02 is equipped with a liquid level transmitter which regulates the flow rate of the stripped effluent (bottoms) to the POTW. Piping is provided to return the stripped effluent to tank VD-15 rather than the POTW if the temperature at the mid-point of the stripping column drops below a given set point, thus indicating that the material does not meet effluent discharge limits. The steam flow rate to the steam stripper is controlled by the overhead temperature of the column. The overhead vapors (approximately 40 percent methylene chloride and 60 percent water) emanating from the top of the packed tower are liquified in a water-cooled condenser and the two-phase distillate is separated by gravity in a decanter. The aqueous upper phase is recycled to the top of the stripping column while the lower layer of methylene chloride is stored in tank VD-04 for reuse. The overhead condenser is vented to the atmosphere by a 2-inch diameter galvanized pipe.

Sampling Program

The on-site sampling program at Facility G took place from August 18 through August 19, 1986. The steam stripping process was evaluated during

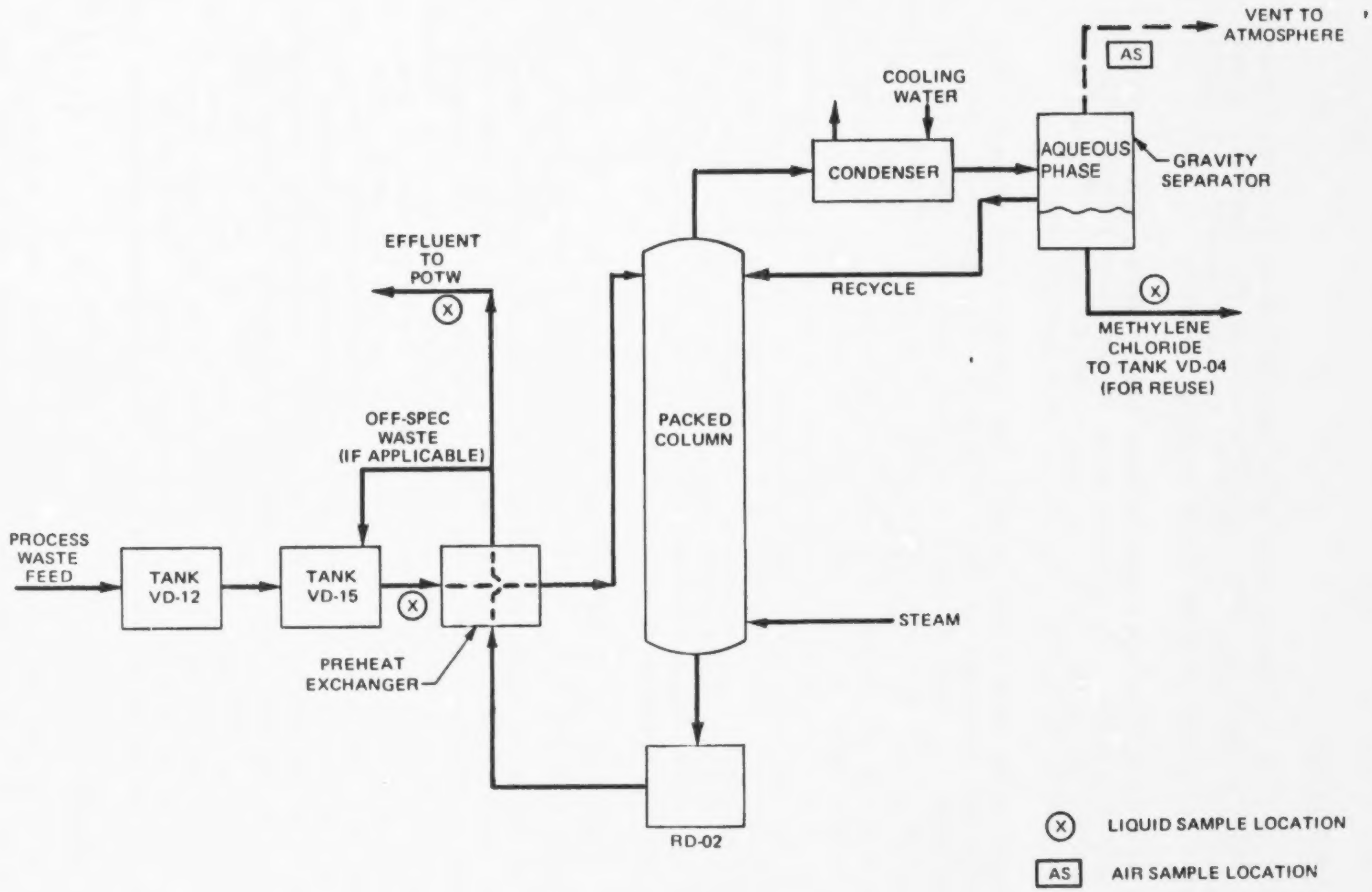
VENT TO ATMOSPHERE
AS
COOLING WATER
CONDENSER
AQUEOUS PHASE
GRAVITY SEPARATOR
EFFLUENT TO POTW
RECYCLE
METHYLENE CHLORIDE TO TANK VD-04 (FOR REUSE)
OFF-SPEC WASTE (IF APPLICABLE)
PACKED COLUMN
PROCESS WASTE FEED
TANK VD-12
TANK VD-15
STEAM
PREHEAT EXCHANGER
RD-02
LIQUID SAMPLE LOCATION
AIR SAMPLE LOCATION

Figure 17. Facility G - methylene chloride steam stripping schematic

the two-day sampling episode by collecting samples of the process waste feed, the treated effluent, the recovered methylene chloride, and the air vent exhaust. Sampling locations are illustrated in Figure 17.

Thirteen discrete grab samples of the process waste feed and treated effluent and 12 air vent exhaust samples were collected over the two-day period at approximately 1-hour intervals. A manually composited sample of the recovered methylene chloride was obtained from the drain hose of the storage tank as the tank was being emptied into a 55-gallon drum. Because the contents of the tank could potentially form several phases, four grab samples were collected as a function of time as the tank contents were discharged. These grabs were blended together and a single sample procured.

The sampling locations and methods are summarized in Table 42. Table 43 summarizes the physical and chemical sample fractions obtained at each sampling point.

Data

Table 44 summarizes the data obtained from the sampling episode at Facility G.

Waste Characterization --

The stripper influent was a free-flowing liquid with predominately one phase. A very thin brown layer was present on top of the samples collected. Flow rate measurements averaged 19.7 l/min for the influent stream. The major components of the influent waste stream were methylene chloride and chloroform at concentrations of 3,600 ppmw and 52 ppmw, respectively. The average suspended solids concentration was measured as 18.9 ppmw.

Product Characterization --

The composite recovered solvent product was a free-flowing liquid with a thin brown layer present on top. The concentration of methylene chloride in the sample was only 5,100 mg/kg which would indicate a relatively low recovery of methylene chloride. At an average flow rate of 0.28 l/min, the recovery of methylene chloride was only 0.088 kg/hr.

The velocity of the vent exhaust from the condenser averaged 100 ft/min. Methylene chloride was the primary noncondensable compound with an emissions rate of 0.095 kg/hr. The total volatile organic emissions averaged 4.3 kg/hr for the two-day period.

Residue Characterization --

The stripped effluent from the steam stripper was a pale yellow, single phase, free-flowing liquid with an average flow rate of 21.8 l/min. The major component in the waste influent, methylene chloride, was reduced to <0.19 ppmw while the level of chloroform, a highly volatile compound, was measured to be 5.3 ppmw.

TABLE 42. FACILITY G - SAMPLE LOCATION DESCRIPTIONS

Sample Location	Description	Type and Time Interval
Process Waste Influent	Grab samples were either discharged directly to the sample containers or first collected in a glass beaker from the sample tap near the preheat exchanger. Samples were collected in individual glass bottles for specific laboratory analysis.	One grab sample hourly
Stripped Effluent	Grab samples were collected in a stainless steel beaker from the sample tap after the preheat exchanger. Samples were distributed to individual glass bottles for specific laboratory analysis.	One grab sample hourly
Recovered Solvent Product	Sample was manually composited from the tank VD-04 drain hose. Samples were distributed to individual glass bottle for specific laboratory analysis.	Composite collected upon draining of tank VD-04
Air Vent	Air samples were collected from the condenser exhaust vent in evacuated glass sampling bulbs.	One grab sample hourly

Table 43. Facility G - physical and chemical sample fractions

Sample Location Description	VOC (1)	Methanol	TOX	TOC/COD	Extractable Organics (1)	Solids & Specific Gravity (1)	%Water/Btu	Viscocity	Temperature	pH
Process Waste Influent	X	X^3	X	X	X^4	X	X^4	X	X	X
Stripped Effluent	X	X^3	X	X	X^4	X			X	X
Recovered Solvent Product	X	X	X	X		X	X	X	X	
Air Vent	X^2								X	

1. Specific compounds identified in Table 2 (Section 3) will be analyzed.
2. Syringe sample to be analyzed for methylene chloride by GC/FID; air bulb samples to be analyzed for compounds in Table 2 (Section 3).
3. A single grab sample per day.
4. Daily composite.

TABLE 44. FACILITY G - ANALYTICAL DATA

Compound	Process Waste Influent		Bottoms Effluent		Overhead Condensate Organic Phase		Removal[d]	Vent	
	Flow Rate (kg/hr)[a]	Concentration (mg/kg)	Flow Rate (kg/hr)[b]	Concentration (mg/kg)	Flow Rate (kg/hr)	Concentration (mg/kg)	(wt. %)	Flow Rate (kg/hr)	Concentration (mg/l)
Methylene Chloride	4.5	3,600	$<2.6 \times 10^{-4}$	<0.19	0.088	5,110	>99.99[c]	0.095	24.7
Chloroform	0.066	52	0.0072	5.3	0.019	1,080	89	0.046	12.0
Carbon Tetrachloride	2.9×10^{-3}	<2.3	$<2.26 \times 10^{-4}$	<0.17	-	-	>92[c]	$<2.30 \times 10^{-4}$	0.06
Total VO	4.6	3,654	7.69×10^{-3}	<5.66	0.107	6,200	>99.8	0.141	36.8
Water	1,255[e]	-	1,360[e]	-	16.9[e]	-	-	4.14	-
Total	1,260	-	1,360	-	17.0	-	-	4.28	-
Additional Parameters									
Volumetric Flows (l/min)	19.7		21.8		0.285			64.3	
Specific Gravity	1.069		1.0425		1.0167			-	

[a] Calculation [(Concentration (mg/l) + (1,069 kg/l)] × Flow Rate (l/hr) (10^{-6}) = kg/hr.

[b] Calculated from material and energy balances using measured flows.

[c] Outlet component concentration is below detection limit, therefore, the percent removal is calculated using the detection limit as the measured concentration.

[d] - Calculated as: (1-(Effluent from Stripper)/(Influent to Stripper)) × 100%

[e] Balance of total flow after accounting for organics.

Evaluation of Effectiveness

Overall the removal of methylene chloride from the waste stream was very effective (>99.99 percent), but the process was less effective (89 percent) at removal of chloroform from the waste stream. Despite the good removal of methylene chloride from the waste stream, the data indicates poor recovery of methylene chloride in the overhead product. The discrepancy in this value could be attributed to the compositing of the solvent product sample. The original sampling plan for Facility G called for use of a COLIWASA to obtain a volume composite sample from the solvent product storage tank since the contents of the tank could potentially form several phases. Due to the equipment setup, it was not possible to use the COLIWASA. Instead, during the time that the tank was discharging, four grab samples were taken from the discharge line. Without being able to see inside the tank, there was no way of knowing whether the composite was an accurate representation of the recovered solvent product. The denser methylene chloride phase, which would be discharged first from the bottom of the tank, may not have been represented in the grab samples.

Residue Management

The bottoms from the methylene chloride stream stripping process are discharged to the local publicly-owned treatment works (POTW).

FACILITY M

Information regarding the stream stripper operations at Facility M was obtained from the report entitled "Field Test and Evaluation of the Steam Stripping Process at Plant H" which was prepared by Research Triangle Institute for EPA-HWERL (December 1986)

Process Description

The steam stripper at Facility M treats a dilute aqueous waste stream which is comprised of wastewater generated from the production of 1,2-dichloroethane and vinyl chloride monomer, wastewaters generated from other parts of the plant and storm water runoff.

The purpose of the steam stripper is to recover 1,2-dichloroethane from plant wastewater for recycle to the production process and to remove volatile organics (VO) prior to wastewater treatment. Figure 18 illustrates the steam stripping process.

Sampling Program

During the two-day sampling episode, five discrete grab samples of the stripper feed, effluent and condensate were taken at 2-hour intervals during the day shift of each day for VO analysis. The condensate separated into an aqueous phase and a heavier organic phase (primarily ethylene dichloride) and samples of both phases were submitted for analysis. Samples for extractable organics, solids and metals were taken each hour during the

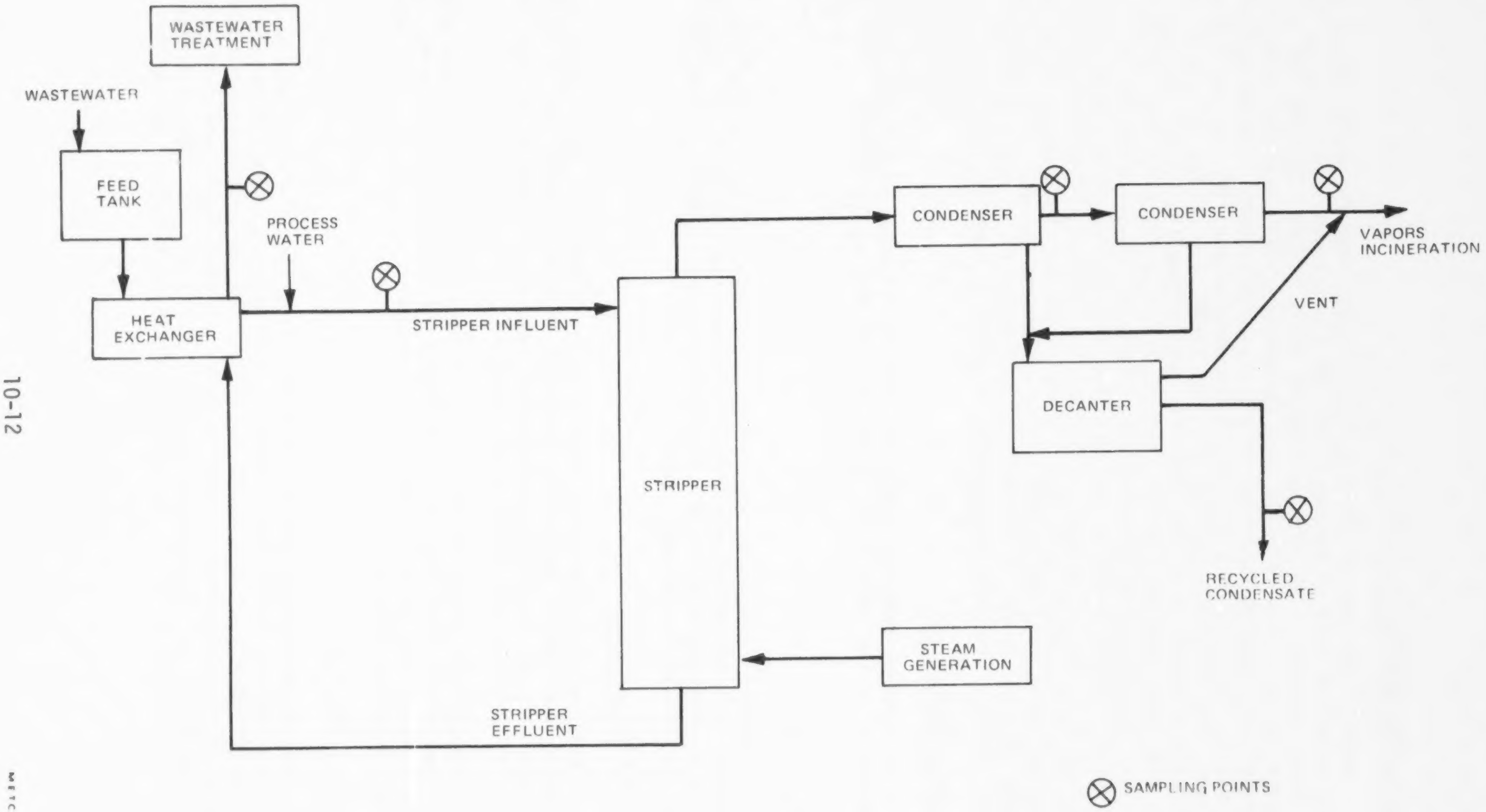
WASTEWATER TREATMENT
WASTEWATER
FEED TANK
HEAT EXCHANGER
PROCESS WATER
STRIPPER INFLUENT
STRIPPER
STRIPPER EFFLUENT
STEAM GENERATION
CONDENSER
CONDENSER
DECANTER
VENT
VAPORS INCINERATION
RECYCLED CONDENSATE
⊗ SAMPLING POINTS

Figure 18. Facility M steam stripping schematic and sampling points.

sampling episode. The extractable organic samples were composited over a 4-hour period to yield two samples per day. The metals and solids samples were composited over an 8-hour period to yield one sample per day. Six discrete grab samples of the condenser vent exhaust were also collected over the two-day period using evacuated electropolished stainless steel canisters.

Data

Table 45 summarizes the data obtained from the sampling episode at Facility M.

Waste Characterization --

As expected the major constituent in the influent stream was 1,2-dichloroethane at a concentration of 5,630 ppmw. The influent also contained chloroform at a concentration of 271 ppmw and measurable traces of 12 other volatile organic compounds. The influent solids concentration was on the order of 0.1 percent. The average flow rate of the influent was measured as 846 l/min.

Product Characterization --

The concentration of 1,2-dichloroethane in the organic phase of the overhead organic condensate averaged 880,000 ppmw and the concentration of chloroform which partitioned into the organic phase averaged 47,000 ppmw. A total of 7 of the 14 constituents found in the wastewater were identified and quantified in the vapors vented from the condenser. Noncondensables that pass through the condensers are collected and vented to an incinerator. The average flow rate of the noncondensable was 186 l/min. Approximately 2.72 kg/hr of volatile organic compounds were vented to the incinerator. The primary component was 1,2-dichloroethane with a mass flow rate of 1.47 kg/hr. Vinyl chloride and chloroform contributed 0.35 and 0.46 kg/hr to the noncondensable compounds flow rate.

Residue Characterization --

Flow rates of the stripper effluent averaged 846 l/min. The steam stripper removed 1,2-dichloroethane to a concentration of 0.097 ppmw. Chloroform was only removed to a concentration of 9.6 ppmw.

Evaluation of Effectiveness

The steam stripper at Facility M had an overall volatile organic removal efficiency of 99.8 percent. This high removal is largely attributed to the 99.998 percent reduction of 1,2-dichloroethane to a concentration of 0.097 mg/kg. The only constituent for which high removals were not consistently attained was chloroform. According to the RTI sampling report, the column experienced tray fouling due to solids accumulation. To reduce solids accumulation, the operator periodically back flushed the column. The tray fouling had an effect on removal efficiencies; but since 1,2-dichloroethane still achieved high removal, the tray fouling is probably not the

TABLE 45. FACILITY M - ANALYTICAL DATA

Component	Influent to Stripper		Effluent from Stripper		Overhead Condensate				Overhead Removal[c] (wt. %)	Vent	
					Aqueous		Organic				
	Flow (kg/hr)	Conc.[a] (ppmw)	Flow (kg/hr)	Conc.[a] (ppmw)	Flow (kg/hr)	Conc.[a] (ppmw)	Flow (kg/hr)	Conc.[a] (ppmw)		Flow (kg/hr)	Conc. (mg/l)
1,2-Dichloroethane	276	5,630	0.0049	0.097	4.3	3,600[e]	210	880,000[e]	99.998	1.47	132
Chloroform	13.3	271	0.48	9.6	0.41	340[e]	11	47,000[e]	96.4	0.46	40.8
Benzene	0.013	0.27	$<5 \times 10^{-4}$	<0.01	-	NA	-	NA	>96	-	-
Carbon Tetrachloride	0.083	1.7	$<5 \times 10^{-4}$	<0.01	-	NA	-	NA	>99.4	-	-
Chlorobenzene	0.019	0.38	$<5 \times 10^{-4}$	<0.01	-	NA	-	NA	>97	-	-
Chloroethane	0.47	9.6	$<5 \times 10^{-4}$	<0.01	-	NA	-	NA	>99.89	0.19	16.8
1,1-Dichloroethane	0.54	11	$<5 \times 10^{-4}$	<0.01	-	NA	-	NA	>99.91	0.05	4.8
1,1-Dichloroethene	0.23	4.7	$<5 \times 10^{-4}$	<0.01	-	NA	-	NA	>99.8	0.16	13.9
1,2-Dichloroethene	0.44	8.9	$<5 \times 10^{14}$	<0.01	-	NA	-	NA	>99.7	0.04	4.0
Methylene Chloride	0.059	1.2	$<5 \times 10^{-4}$	<0.01	-	NA	-	NA	>99.2	-	-
Tetrachloroethene	0.069	1.4	$<5 \times 10^{-4}$	<0.01	-	NA	-	NA	>99.3	-	-
1,1,2-Trichloroethane	0.37	7.5	$<5 \times 10^{-4}$	<0.01	-	NA	-	NA	>99.9	-	-
Trichloroethene	0.24	4.8	$<5 \times 10^{-4}$	<0.01	-	NA	-	NA	>99.8	-	-
Vinyl Chloride	0.41	8.4	$<5 \times 10^{-4}$	<0.01	-	NA	-	NA	>99.9	0.35	31.7
Total VO	292	5,960	0.50	<9.8	4.7	3,900	220	927,000	>99.8	2.72	244
Water	48,668[g]	-	50,760	-	1,200	-	20	-	-	-	-
Total	48,960[h]	-	50,760	-	1,200	-	240	-	-	-	-
Additional Parameters											
Volumetric Flow Rate (l/min)	816	-	846	-	20.3 l	-	3.24	-	-	186	-
Specific Gravity	1.0	-	1.0	-	-	-	1.24	-	-	-	-
Average Temperature (°C)	84		108				34.00				

NA - Not analyzed for this component.

[a] Average of concentrations measured during testing (10 grab samples).

[c] Calculated as : (1 - (Effluent from stripper)/(Influent to stripper)) x 100%.

[d] Sample calculation: (Average component concentration, ppmw) x (Average flow rate) x $(10^6 \text{ ppmw})^{-1}$ = Component flow rate.

[e] Only chloroform and 1,2-dichloroethane were analyzed in the condensate. Because of the use of average flows and aveage concentrations, the component mass balance for these components may not be close as was usually obtained at a given sampling time.

[f] All concentrations were below detection limit. One-half of limit used for calculation purposes.

[g] Balance of flow after accounting for organics.

[h] Average of flow rates measured during testing.

major factor in poor chloroform removal. The EPA Office of Water explained that the poor removal was due to the decomposition of chloral (C_2HCl_3O), which is formed in the production process, to form chloroform. To increase the removal efficiency for chloroform under these conditions, the pH should be maintained consistently above 11. During the sampling episode, the influent stream average pH was 6.5, the bottoms effluent average pH was 8.4 and the condensate average pH was 6.2, thus the low removal efficiency was observed.

To reduce any adverse affects that tray fouling has on volatile organic removal efficiencies, a solids removal step prior to steam stripping the waste could be implemented. Since the sludge generated from the solids removal step may result in a disposal problem and also become an additional source of volatile organic emissions, the facility feels it is more economical to clean the existing system periodically than to install an additional solids removal, treatment and disposal system.

Residue Management

The bottoms effluent from the steam stripper passes through a heat exchanger to effect some cooling and to preheat the stripper feed stream. After passing through a second heat exchanger, the effluent is sent to the wastewater treatment system. Noncondensables released through the condenser vent flow to an off gas incinerator.

FACILITY N

Information regarding the steam stripper operations at Facility N was obtained from the report entitled "Field Test and Evaluation of the Steam-Stripping Process at Plant I" which was prepared by Research Triangle Institute for EPA-HWERL (March 1987).

Process Description

Facility N produces mono-carbon chlorinated solvents such as methylene chloride, chloroform and carbon tetrachloride. The steam stripper treats a dilute aqueous waste stream consisting of equipment washwater and rainfall collected from diked areas around the plant.

The recovery of methylene chloride, carbon tetrachloride and chloroform to recycle to the production process is the primary objective of this steam stripper. The stripper also reduces the concentration of total volatile organic compounds to enable the effluent stream to be discharged to the river under an NPDES discharge permit. Figure 19 illustrates the steam stripping process at Facility N. A detailed process description is presented in the RTI report.

Sampling Program

During the two-day sampling episode, samples of the stripper influent, bottoms and condensate were taken at approximately 2-hour intervals during the day shift for each day of the two-day test (a total of 11 samples) for

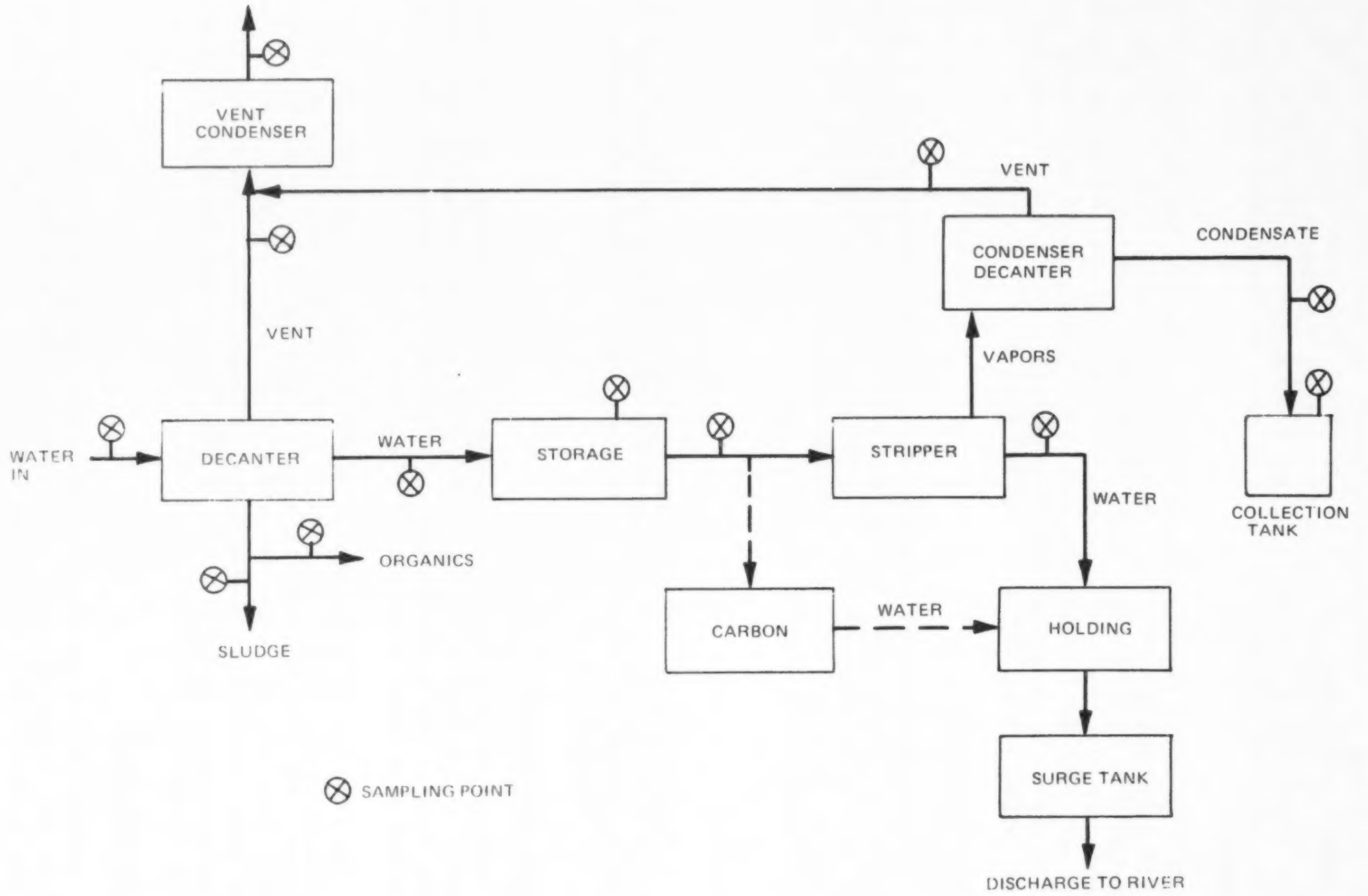
VENT CONDENSER
VENT
CONDENSER DECANTER
CONDENSATE
VENT
VAPORS
WATER IN
DECANTER
WATER
STORAGE
STRIPPER
WATER
COLLECTION TANK
ORGANICS
SLUDGE
CARBON
WATER
HOLDING
SURGE TANK
DISCHARGE TO RIVER
SAMPLING POINT

Figure 19. Facility N steam stripping schematic and sampling points.

VO analysis. Samples for headspace analysis were also taken at these sampling locations. Composite samples of the influent and bottoms streams were analyzed for filterable and dissolved solids. A metals analysis was also performed using the composite influent sample. The pH and temperature of the liquid samples were also taken several times during each day of the sampling episode.

The influent to the solids decanter was sampled throughout the first day and analyzed for VO filterable and dissolved solids, metals and VO in the headspace. Samples of the decanted organic and aqueous layers were analyzed for all of the same parameters except metals. The sludge generated from the decanting process was sampled to analyze for VO, total solids, VO in headspace and also for analysis by EPA's Toxicity Characteristic Leaching Procedures (TCLP).

Vapor samples were taken at the primary condenser vent, secondary condenser vent and the stripper feed tank vent three times each test day. Vapor samples were also collected over the open organic collection tank and the decanter vent. All vapor samples from the condensers were taken in evacuated electropolished stainless steel canisters.

Data

Table 46 summarizes the data obtained from the sampling episode at Facility N.

Waste Characterization --

The influent flow rate to the stripping process was approximately 40.7 liters/min. High concentrations of methylene chloride and chloroform, 4,490 mg/kg and 1,270 mg/kg, respectively, were detected in the stream. The pH of the influent waste stream was 5 during day one but increased to 11.5 during day two. This increase was due to the treatment of a batch of wastewater which was transferred from the solids decanter with a very high pH after caustic addition.

Product Characterization --

The overhead organic condensate flow rate averaged 0.14 l/min. The recovered methylene chloride and chloroform concentrations were 1,180,000 and 319,000 mg/l, respectively.

Residue Characterization --

Total VO in the effluent stream was reduced to below 0.037 mg/kg at an effluent flow rate of approximately 44.7 l/min. On an average basis, all compounds except for methylene chloride and chloroform were removed to below their detection limit. For methylene chloride, 4 of 11 discrete grabs were below the detection limit which when averaged with the seven remaining samples produces a concentration of 0.011 mg/kg. This concentration is reported as <0.011 mg/kg since the actual detection limit was used to calculate the average concentration and the concentration for those four samples

TABLE 46. FACILITY N - ANALYTICAL DATA

Compound	Stripper Influent		Stripper Effluent		Organic Condensate[b]		Percent Removal (wt. $)	Primary Condenser Vent Flow (kg/hr)
	Flow (kg/hr)[a]	Conc. (mg/kg)	Flow (kg/hr)	Conc. (mg/kg)	Flow (kg/hr)	Conc. (mg/l)		
Chloromethane	0.0796	35	$<1.35 \times 10^{-5}$	<0.005	-	-	>99.98[c]	0.0086
Methylene Chloride	10.96	4,490	$<2.88 \times 10^{-5}$	<0.011	9.42	1,180,000	>99.999[c]	1.05
Chloroform	3.19	1,270	$<1.67 \times 10^{-5}$	<0.006	2.78	319,000	>99.999[c]	0.160
Carbon Tetrachloride	0.316	55	$<1.35 \times 10^{-5}$	<0.005	-	-	>99.99[c]	0.014
Trichloroethylene	.028	5.6	$<1.35 \times 10^{-5}$	<0.005	-	-	>99.9[c]	-
1,1,2-Trichloroethane	.013	5.3	$<1.35 \times 10^{-5}$	<0.005	-	-	>99.9[c]	-
Total VO	14.6	5,860	$<9.95 \times 10^{-5}$	<0.037	12.2	1,499,00	>99.999[c]	1.23
Water	2,425	-	2,680	-	-	-	-	0.013
Additional Parameters								
Flow Rates (l/min)	40.7	-	44.7	-	0.14	-	-	57
Specific Gravity	1.0	-	1.0	-	-	-	-	-

[a] Calculation: Concentration (mg/l) * Overall Flow Rate (l/hr) x 10^{-6} = kg/hr.

[b] Condensate rate estimated from organics in with feed minus organics out with uncondensed vapor and bottoms.

[c] Outlet component concentration is below detection limit, therefore, the percent removed is calculated using the detection limit as the measured concentration.

[d] Calculated as: (1-(Effluent from Stripper)/(Influent to Stripper)) x 100$

may very well have been below the detection limit concentration. The effluent pH during day one was 5 and increased to 11.4 during day two.

Evaluation of Effectiveness

Referring to Table 46 it can be seen that the steam stripper reduced the total VO concentration in the waste stream by five orders of magnitude and attained a 99.999 percent removal efficiency. The total VO was reduced from 5,860 mg/kg to <0.037 mg/kg. Chloroform had a 99.999 percent removal efficiency at this facility whereas at Facility M the removal of chloroform was relatively low.

The organic condensate was highly concentrated in the recovered methylene chloride (1,180,000 mg/l). The vapor flow rate from the condenser was 57 l/min (1.23 kg/hr). The noncondensable vapor consisted primarily of methylene chloride (1.05 kg/hr). The condenser efficiency for methylene chloride was approximately 90 percent. The overall efficiency of the condenser was also approximately 90 percent.

The steam stripper at Facility N demonstrated an efficient recovery of mono-chloronated solvents and substantial reduction of volatile organics in the effluent stream.

Residue Management

The effluent from the stripper is cooled by a heat exchanger and is pumped to one of two open-topped holding tanks (about 19,000 L or 5,000 gal.) where it is analyzed to check if the stream meets discharge permit limits. The current NPDES discharge limits for methylene chloride, carbon tetrachloride and chloroform are 50, 55 and 75 parts per billion (ppb), respectively. If the stream does meet the limits as was the case during the test period, the pH is adjusted for final discharge to the river under the NPDES permit. If the water does not meet permit limits, the stream is treated again by either the steam stripper or the granular activated carbon adsorption unit. The granular activated carbon adsorption system is considered a backup unit and is used in place of the stripper in the event the stripper is down or if the capacity of the stripper is exceeded, therefore, it is not used as a polishing step.

SECTION 11

ACTIVATED SLUDGE TREATMENT

THEORY

Biological treatment processes destroy organic compounds in aqueous waste by biologically catalyzed oxidation. In the activated sludge process, which is the most common form of aerobic biological treatment, aerobic microorganisms suspended in the wastewater metabolize dissolved organics, converting them to carbon dioxide, water and biomass.

Figure 20 is a schematic diagram illustrating the activated sludge process. After primary treatment (screening, grit removal and primary sedimentation), the feed or influent wastewater is continuously pumped into a large open basin or tank in which aerobic microorganisms are suspended at a concentration of 1500 to 3000 mg/l (as volatile suspended solids). The tank is aerated to provide agitation and oxygen for the biological oxidation reactions. Typical aeration tank retention times are from four to eight hours. Effluent from the aeration tank flows to a secondary clarifier for gravity settling of the biological sludge. A portion of the sludge is wasted and the remaining sludge is returned to the aeration basin to maintain an optimum biomass concentration and a solids retention time or sludge age of 5 to 15 days.

If the wastewater is deficient in nitrogen or phosphorus, these nutrients must be added to maintain optimum biological activity. Temperature and pH must be maintained within ranges suitable for the microorganisms to live.

The principal waste generated by the activated sludge process is the waste sludge. This material is typically dewatered in a filter press, rotary vacuum filter or centrifuge prior to land disposal or incineration of the residue.

PROCESS DESCRIPTION

Facility I manufactures plastic adhesives, UV absorbers (organic chemicals used in plastics as light stabilizers), surfactants and animal feed intermediates. As many as 95-105 different batch wastewater streams generated from 25 unit processes contribute to the influent flow of the on-site biological wastewater treatment plant. Specific to the objectives of the test program, one of the identified waste streams is the bottom waters from a continuously operated methanol recovery still.

Two separate waste streams are treated by the biological wastewater treatment plant. The first stream contains the majority of the wastewater (about 3.2 million gallons per day (mgd)) from the manufacturing processes. The methanol still bottoms waste stream, which is characterized as a RCRA F003 waste stream, constitutes about 0.1 percent of the 3.2 mgd. The

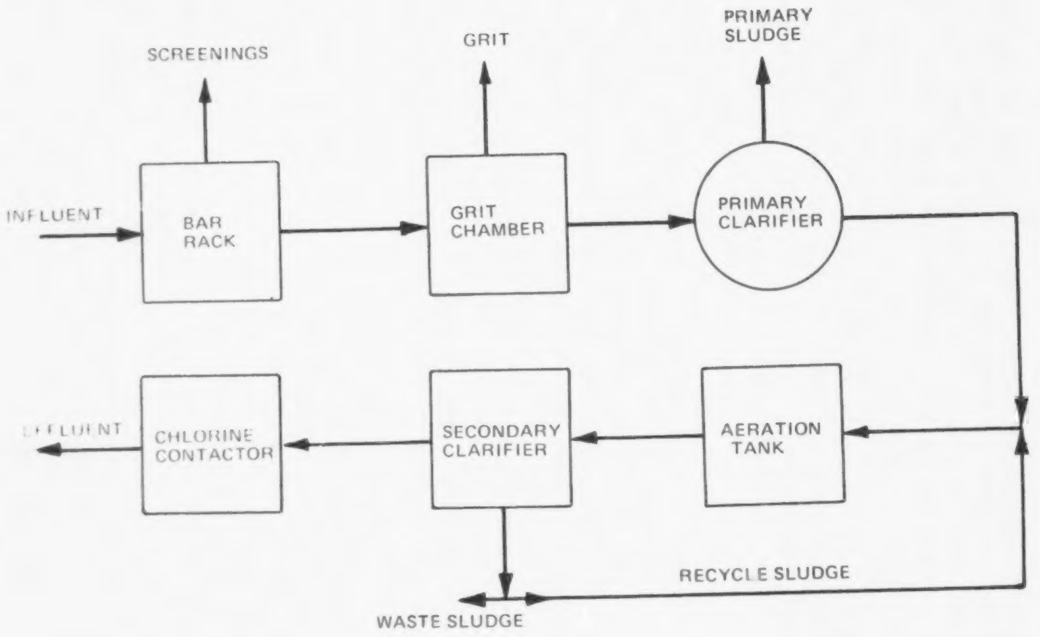
SCREENINGS
GRIT
PRIMARY SLUDGE
INFLUENT
BAR RACK
GRIT CHAMBER
PRIMARY CLARIFIER
EFFLUENT
CHLORINE CONTACTOR
SECONDARY CLARIFIER
AERATION TANK
RECYCLE SLUDGE
WASTE SLUDGE

Figure 20. Activated sludge process schematic.

methanol waste stream is comprised of approximately 99.5 percent water and 0.5 percent methanol. Other sources of methanol which also contribute low concentrations of methanol to the 3.2 mgd waste stream include: methanol recovered in the drying of finished products by air or vacuum stripping, and additional wastewaters dilute in methanol and not economically recoverable. The second waste stream is from the animal feed intermediate (AFI) operation which produces about 300,000 gpd of a high BOD_5, nutrient rich waste.

The biological treatment plant treats 3.5 mgd of wastewater streams generated on-site. Figure 21 presents the flow schematic and sampling locations for the treatment system. The wastewater flows through a mechanically cleaned bar rack and then to a neutralization tank where lime is added to maintain a pH between 7.0 and 8.5. The neutralized waste flows by gravity to a 100-foot diameter (700-gallon capacity) primary clarifier. The primary sludge is pumped to the primary sludge gravity thickener about once per week. During the sampling episode, sludge was continuously pumped to prevent the pump and the clarifier raking mechanism from clogging. The solids concentration of the sludge is about 0.3 percent, when removed once per week. However, with the continuous pumping of sludge from the clarifier, the solids content of the sludge is extremely low with some visible oil, grease, and settleable solids present. The overflow from the primary gravity thickener is returned to the neutralization tank at the head of the treatment plant. The thickened primary sludge is mixed with alum (0.05 lbs. of alum/lb of dry solids) and lime (0.7 lbs. of lime/lb of dry solids) in a sludge conditioning tank and then dewatered in a low pressure vacuum filter. The dewatered solids content from the vacuum filter is about 17 percent by weight. Facility I estimates that the dewatered sludge from the vacuum filter press constitutes 10 percent of the total residues generated. Filtrate from the sludge dewatering process is pumped to the aeration basin and the dewatered primary sludge is placed in a RCRA surface impoundment adjacent to Facility I.

The primary effluent flows by gravity to a 26 million gallon capacity earthen-lined equalization basin with an estimated 12 to 24 hour retention time. From the lagoon, the wastewater is pumped to a 6-million gallon capacity aeration basin. The aeration basin has a hydraulic retention time of 41 hours. The flow to the aeration basin from the equalization lagoon is constant at approximately 3.2 mgd. The AFI waste is discharged directly to the aeration basin. The aeration basin is equipped with six 125-hp fixed basin platform aerators. The use of the aerators is staggered with only five aerators operating at one time to maintain the dissolved oxygen content at about 2.5 mg/l. Approximately 55 gallons of phosphoric acid (37 percent in solution) is added to the aeration basin per day as a nutrient supplement. Facility I has not had any upsets at the biological plant for several years.

Cationic polymer and alum are added to the effluent from the aeration basin which flows to a splitter box and then to one of three secondary clarifiers. At the current waste loading, two of the three clarifiers are used. The clarifiers are each 70 feet in diameter with a 12-foot side water depth. The settled sludge is rapidly removed by draft tubes. A portion of the sludge is returned to the aeration basin (return activated sludge) and a

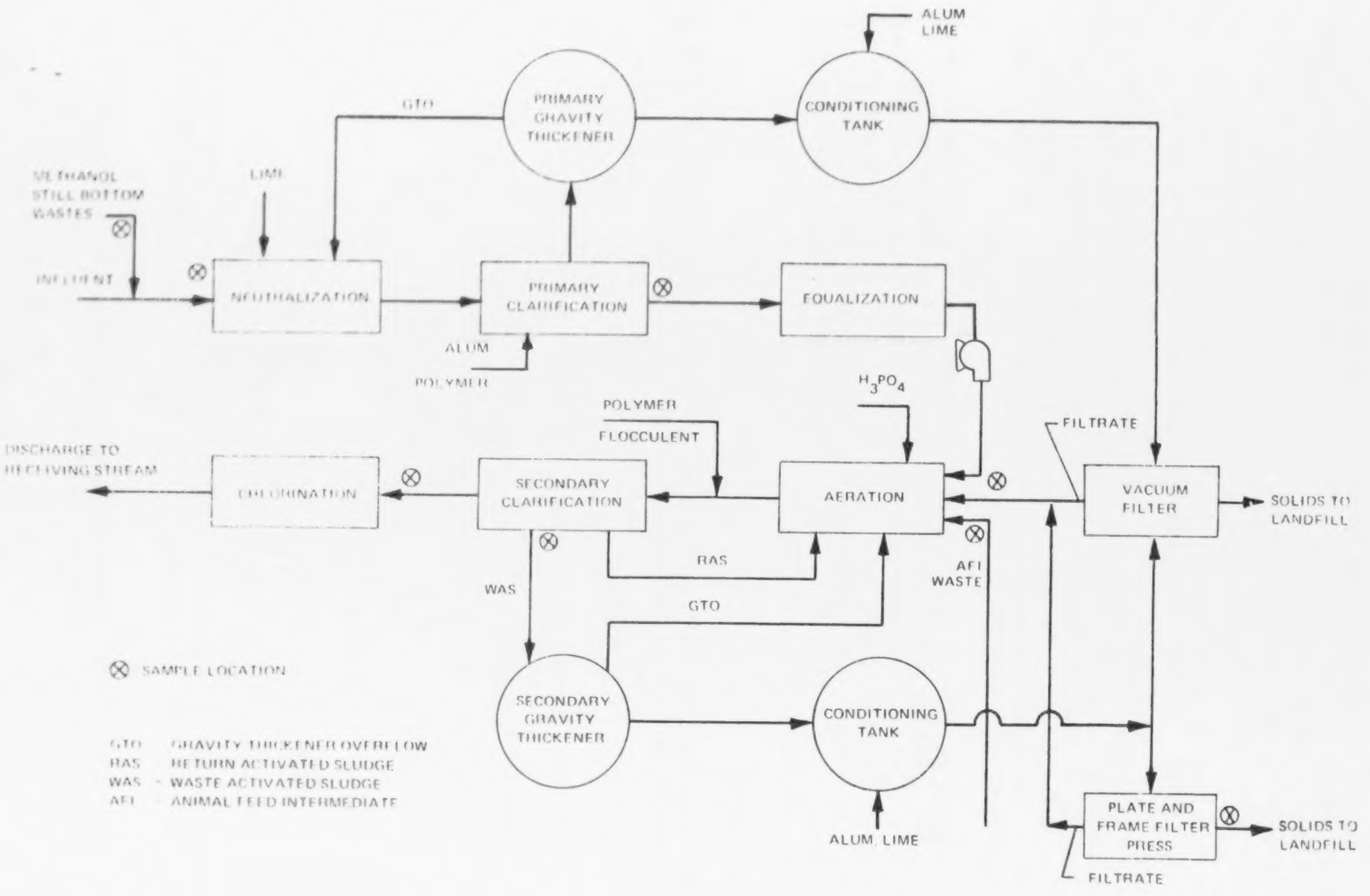
ALUM
LIME
GTO
PRIMARY GRAVITY THICKENER
CONDITIONING TANK
METHANOL STILL BOTTOM WASTES
LIME
INFLUENT
NEUTRALIZATION
PRIMARY CLARIFICATION
EQUALIZATION
ALUM
POLYMER
H_3PO_4
POLYMER
FLOCCULENT
FILTRATE
DISCHARGE TO RECEIVING STREAM
CHLORINATION
SECONDARY CLARIFICATION
AERATION
VACUUM FILTER
SOLIDS TO LANDFILL
RAS
AFI WASTE
WAS
GTO
SAMPLE LOCATION
SECONDARY GRAVITY THICKENER
CONDITIONING TANK
GTO - GRAVITY THICKENER OVERFLOW
RAS - RETURN ACTIVATED SLUDGE
WAS - WASTE ACTIVATED SLUDGE
AFI - ANIMAL FEED INTERMEDIATE
PLATE AND FRAME FILTER PRESS
SOLIDS TO LANDFILL
ALUM, LIME
FILTRATE

Figure 21. Biological wastewater treatment plant - Facility I.

portion is wasted to the sludge dewatering operation (waste activated sludge). Return activated sludge rates from the secondary clarifier vary from 30-50 percent of the incoming flow based on maintaining a mixed liquor suspended solids (MLSS) concentration between 3,000 and 4,000 mg/l. The waste activated sludge is lifted by piston pumps to the secondary gravity thickener. The secondary gravity thickener overflow is returned to the aeration basin. The thickened waste activated sludge is conditioned with alum and lime and is dewatered by either the plate and frame filter press or the vacuum filter. As mentioned earlier, the dewatered solids content from the vacuum filter is about 17 percent by weight; the dewatered solids from the plate and frame filter press is about 40 percent by weight. Facility I personnel estimate that the dewatered sludge from the plate and frame filter press constitutes about 90 percent of the total residues generated. The dewatered sludge from the vacuum filter press constitutes the remaining 10 percent. The dewatered waste activated sludge is placed in a RCRA surface impoundment adjacent to Facility I.

Filtrate from the waste activated sludge dewatering operation is returned to the aeration basin. The treated effluent from the secondary clarifiers is combined and then flows by gravity to a chlorine contact tank. The treated effluent is dosed with chlorine (residual of 0.6-0.8 ppm of chlorine) year-round. The detention time through the chlorine contact tank is 12-15 minutes at a flow of 3.5 mgd. The secondary effluent is then discharged to a nearby river tributary.

Sampling Program

The biological treatment process was evaluated by collecting samples at the following locations as indicated by Figure 21 and described in Table 47.

- methanol still bottoms
- influent to neutralization
- primary clarifier effluent
- influent to aeration basin
- AFI waste
- secondary clarifier effluent
- waste activated sludge
- dewatered solids from plate and frame filter press

Three discrete samples (grab #1, grab #2, and grab #3) were collected at 0000 hr, 0800 hr and 1600 hr respectively each day for three consecutive days.

In addition, 3-24 hr composite samples were collected concurrently at the following locations (refer to Figure 21).

TABLE 47. FACILITY I SAMPLING LOCATIONS AND PROCEDURES

Sample Description	EPA Sample Number	Sample Location	Sampling Procedure
Methanol Still Bottoms		Samples were procured from a sample tap on the discharge line of the methanol recovery process.	The sample tap was purged prior to collecting each discrete sample. The methanol still bottoms were collected in a 5 gallon capacity stainless steel container. A glass pyrex beaker was then used to distribute the sample to the respective sample containers. To ensure a representative sample each container was first filled to one sixth of its volume and this procedure was then repeated six times. VOC and methanol vials were filled to one sixth of its volume and this procedure was then repeated six times. VOC and methanol vials were filled individually and the oil and grease sample was taken directly from the sample tap. Three discrete grab samples were collected at 8-hour intervals and each discrete sample was analyzed separately.
Influent to Neutralization		Samples were taken from the 36-inch diameter influent pipe to the treatment plant, immediately upstream of the plant headworks.	A continuous 24-hour composite sample was collected by an ISCO sampler with aliquots automatically collected every 20 minutes. The composite sample was fractioned into specific bottles for extraction organics, metals, Biochemical Oxygen Demand (BOD_5), Chemical Oxygen Demand (COD), Total Organic Carbon (TOC), and solids analyses. Grab composite samples for volatile organic compounds (VOC), methanol and Total Organic Halides (TOX) were manually collected every four hours using a pre-rinsed pyrex beaker attached to a sampling pole. These samples were composited in the lab for the analyses. Discrete grab samples for VOC and methanol were collected manually every eight hours with a pre-rinsed pyrex beaker attached to a sampling pole. These samples were analyzed discretely by the lab.

TABLE 47 (Continued). FACILITY I SAMPLING LOCATIONS AND PROCEDURES

Sample Description	EPA Sample Number	Sample Location	Sampling Procedure
Primary Clarifier		Samples were collected from the outlet stream of the primary clarifier prior to the stream flowing by gravity to the equalization basin. The ISCO sampler tubing was placed at mid-depth of the waste stream and secured to face into the waste stream by means of a rigid aluminum conduit.	A continuous 24 hour composite sample was collected by an ISCO sampler with aliquots automatically collected at 20 minute intervals. The composite sample was fractioned into specific bottles for extractable organics, metals, BOD_5, COD, TOC, and solids analyses. Grab composite samples for VOC, methanol, TOX and O&G were manually collected every four hours using a pre-rinsed pyrex beaker attached to a sampling pole. These samples were composited in the lab for the analyses. Discrete grab samples for VOC and methanol were collected manually every eight hours with a pre-rinsed pyrex beaker attached to a sampling pole. These samples were analyzed discretely by the lab.
Aeration Basin Influent		Discrete and composite samples were collected from the equalization basin which feeds into the aeration basin. The ISCO sampler tubing was suspended by means of an attached stainless steel weight.	A continuous 24-hour composite sample was collected by an ISCO sampler with aliquots automatically collected every 20 minutes. The composite sample was fractioned into specific bottles for extractable organics, Biochemical Oxygen Demand (BOD_5), Chemical Oxygen Demand (COD), Total Organic Carbon (TOC), and solids analyses. Grab composite samples for Volatile Organic Compounds (VOC), methanol, Total Organic Halides (TOX) and Oil and Grease (O&G) were manually collected every four hours using a pre-rinsed pyrex beaker attached to a sampling pole. These samples were composited in the lab for the anaylses. Discrete grab samples for VOC, methanol, extractable organics, TOX, metals, BOD_5, COD, TOC, solids and O&G were collected manually every 8 hours with a pre-rinsed pyrex beaker attached to a sampling pole. These samples were analyzed discretely by the lab.

TABLE 47 (Continued). FACILITY I SAMPLING LOCATIONS AND PROCEDURES

Sample Description	EPA Sample Number	Sample Location	Sampling Procedure
Secondary Clarifier Effluent		Samples were taken at the influent location of the chlorination tank, prior to the point where chlorine is introduced.	A continuous 24-hour composite sample was collected by an ISCO sampler with aliquots automatically collected every 20 minutes. The composite sample was fractioned into specific bottles for extractable organics, metals, BOD_5, COD, TOC, solids, soluble nitrogen and soluble phosphorous and solids analyses. Grab composite samples for VOC, methanol, TOX, and O&G were manually collected every four hours using a pre-rinsed pyrex beaker attached to a sampling pole. These samples were composited in the lab for the analyses. Discrete grab samples for VOC, methanol, extractable organics, TOX, metals, BOD_5, COD, TOC, solids and O&G were collected manually every eight hours with a pre-rinsed pyrex beaker attached to a sampling pole. These samples were analyzed discretely by the lab.
Waste Activated Sludge		Grab samples were collected from a sample tap located off the secondary sludge pump. The sludge was pumped from the secondary clarifier to the gravity thickener.	The sample tap was purged prior to collecting each discrete sample. Grab samples were taken directly from the tap at eight hour intervals and were analyzed for TOX, metals, solids, alkalinity/acidity and Toxicity Characteristic Leaching Procedure (TCLP).
Dewatered Sludge		Samples were taken from the conveyor on the discharge side of the plate and frame filter press.	One discrete grab per day was collected during the sampling episode using a stainless steel trowel and bucket. Samples were analyzed for metals, solids, alkalinity/acidity and TCLP.
Primary Sludge		Samples were taken from a tap located on the primary sludge pump located at the center of the primary clarifier.	One discrete grab sample was collected per day during the sampling episode. Samples were taken directly from the sample tap and were analyzed for TOX, metals, solids, alkalinity/acidity and TCLP.

TABLE 47 (Continued). FACILITY I SAMPLING LOCATIONS AND PROCEDURES

Sample Description	EPA Sample Number	Sample Location	Sampling Procedure
AFI Waste		Samples were taken from the discharge stream that diverts the AFI waste directly into the aeration basin. This sampling point was immediately upstream of the parshall flume used to monitor the flow of the AFI waste stream.	A continuous 24-hour composite sample was collected by an ISCO sampler with aliquots automatically collected every 20 seconds. After 24 hours the composite sample was fractioned into specific bottles for extractable organics, metals, BOD_5, COD,TOC and solids analyses. Grab composite samples for VOC, methanol and TOX were manually collected every four hours using a pre-rinsed pyrex beaker. Oil and grease samples were taken directly from the waste stream. These grab composite samples were composited in the lab for the analysis. Discrete grab samples for VOC, methanol, extractable organics, TOX, metals, BOD_5, COD, TOC and solids were collected manually every eight hours with a pre-rinsed pyrex beaker. The discrete grab for oil and grease analysis was taken directly from the waste stream. These grab samples will be analyzed discretely by the lab.
Aeration Basin		The mixed liquor samples were a composite of six grabs (three samples along the two catwalks) at points between the surface aerators. The effluent from the aeration basin was also sampled for total suspended solids.	The aeration basin was sampled once per day using a stainless steel bucket to composite the six grab samples. The composite samples were analyzed for mixed liquor suspended solids (MLSS) and mixed liquor volatile suspended solids (MLVSS). The dissolved oxygen (DO) readings were taken at the sampling locations. Samples were collected with a stainless steel bucket from the aeration basin effluent. The effluent samples were analyzed for total suspended solids (TSS) and settleable solids to determine the sludge volume index (SVI).
Background Samples Sampler Blank			Distilled water was run through each of the automatic samplers and collected as a composite sampler blank.

- process waste influent
- influent to neutralization
- influent to aeration basin
- AFI waste
- secondary effluent

DATA

Waste Characterization

The principal reason for selection of Facility I for this sampling program was that the wastewater treated at the facility includes a RCRA F003 waste - still bottoms from direct steam distillation of spent methanol. Therefore, the concentration of methanol in the F003 waste stream itself and in the activated sludge process samples is of primary interest. Significant data characterizing process grab and 24-hr composite samples are summarized in Tables 48-57.

Methanol Still Bottoms --

Methanol is the only volatile organic compound found at significant concentration in the methanol still bottom waste stream. Methanol concentration in nine grab samples collected during the three-day sampling program ranged from 350,000 μg/l to 1,600,000 μg/l. The calculated average methanol concentration is 697,778 μg/l, with a standard deviation of 461,345 μg/l.

One semivolatile compound, 2-chlorophenol, was detected at concentrations up to 120 μg/l in some of the grab samples. Only one toxic metal, zinc, was measured above 1 mg/l in any methanol still bottoms sample. Its concentration ranged from 2 to 7 mg/l and averaged 5.1 mg/l. The pH of the discrete grab samples ranged from 4.0 to 5.5. The 5-day biochemical oxygen demand (BOD_5) of the wastewater grab samples ranged from 267 to 1280 mg/l with an average value of 525 mg/l. BOD_5 appears to be directly proportional to methanol concentration, with a BOD_5/methanol ratio of approximately 0.75 for all samples. Methanol is clearly the major organic compound contributing to the BOD_5. Total organic carbon (TOC) values are very high relative to methanol and have no consistent relationship to the BOD_5 values. Oil and grease levels were highly variable, ranging from 124 to 8,480 mg/l in the nine grab samples.

Influent to Aeration --

Several volatile compounds were found in measurable concentrations in the influent to aeration grab samples. Methylene chloride concentrations ranged from 29 to 240 μg/l with a mean value of 84 μg/l. 2-propanone concentrations ranged from 25 μg/l to 1100 μg/l with a mean value of 774 μg/l. 2-butanone was found in concentrations of <10 μg/l to 390 μg/l with a mean

TABLE 48. FACILITY I -- SUMMARY OF ANALYTICAL DATA OF DISCRETE SAMPLES

Pollutant	Units	Calc. Average Meth. Still Bottoms	Calc. Average Influent to Aeration	Calc. Average AFI Influent	Calc. Average Secondary Effluent	Calc. Average Waste Act. Sludge
VOLATILE ORGANIC COMPOUNDS						
methylene chloride	ug/l	7.3	84	40	33	537 *
acetone (2-propanone)	ug/l	19	774	<113	49	84 *
2-butanone	ug/l	<10	131	<35	47	22 *
4-methyl-2-pentanone	ug/l	<10	2,557	<34	47	15 *
toluene	ug/l	<5	1,459	<18	24	<5 *
chlorobenzene	ug/l	<5	540	<18	24	<5 *
methanol (C)	ug/l	697,778	56,667	<10,000	<10,000	<10,000 *
SEMI-VOLATILE ORGANIC COMPOUNDS						
2-chlorophenol	ug/l	75	22	<22	22	55 *
PRIORITY POLLUTANT METALS						
zinc	mg/kg	5.1	39	<13	17	1033 *
OTHER ANALYSES						
BOD_5	mg/l BOD	525	120	1,396	9.8	
TOC	mg/l	4198	99	1040	13	
oil & grease	mg/l	2174	28	443	55	
pH	pH units	4.4	5.9	8.3	6.4	6.5

* = Units are ug/kg.
Analyte concentrations below detection limits were considered at the detection limit for calculations of averages.

TABLE 49. FACILITY I INFLUENT TO AERATION GRAB SAMPLES -- DAY 1

Pollutant	Units	Day 1-Grab#1 Influent to Aeration 13111	Day 1-Grab#2 Influent to Aeration 13112	Day 1-Grab#3 Influent to Aeration 13113	Day 1-Grab Average Influent to Aeration	Day 1-24 Hr. Comp Influent to Aeration 13114
VOLATILE ORGANIC COMPOUNDS						
methylene chloride	ug/l	73	29	67 B	56	84
acetone (2-propanone)	ug/l	1,100 B	25 B	<840 B	655	890
2-butanone	ug/l	390	<10	130	177	180
4-methyl-2-pentanone	ug/l	2,900	<10	2,800	1,903	3,300
toluene	ug/l	450	<5	1,100	518	1,000
chlorobenzene	ug/l	57	<5	310	124	1,000
methanol (C)	ug/l	120,000	90,000	90,000	100,000	130,000
SEMI-VOLATILE ORGANIC COMPOUNDS						
2-chlorophenol	ug/l	<20	<20	<20	<20	20
PRIORITY POLLUTANT METALS						
zinc	mg/kg	8	7	6	7	50
OTHER ANALYSES						
BOD_5	mg/l BOD	139	124	129	131	169
COD	mg/l	I	I	I	I	I
TOC	mg/l	70	80	77	76	78
oil & grease	mg/l	54	43	23	40	23
pH	pH units	6.0	6.0	6.0	6.0	6.0

A = Sample had both liquid and semi-solid phases. TOC is calculated as the weighted average of the two phases and is expressed as ppm on a weight basis.

B = Analyte found in blank as well as in sample. Value has not been corrected for the blank contamination but the concentration of the analytes found in the lab blanks are given below.
13111, 13112, 13113, 13114; acetone in lab blank = 14 ug/l
13113; methylene chloride in lab blank = 3.0 ug/l
13113; benzene in lab blank = 1.2 ug/l

(C) = Evaluated by gas chromatograph/flame ionization detector.
I = Interference - All samples reduce $K_2Cr_2O_7$ prior to titration.
NA = Not analyzed.
* = Units are ug/kg
Analyte concentrations below detection limit were considered at detection limit for calculation of averages.

TABLE 50. FACILITY I INFLUENT TO AERATION GRAB SAMPLES -- DAY 2

Pollutant	Units	Day 2-Grab#1 Influent to Aeration 13138	Day 2-Grab#2 Influent to Aeration 13139	Day 2-Grab#3 Influent to Aeration 13140	Day 2-Grab Average Influent to Aeration	Day 2-24 Hr. Comp Influent to Aeration 13141
VOLATILE ORGANIC COMPOUNDS						
methylene chloride	ug/l	240	100	70	137	84
acetone (2-propanone)	ug/l	690 B	1,000	1,000	897	880
2-butanone	ug/l	120	240	<33	131	<50
4-methyl-2-pentanone	ug/l	2,700	3,000	1,800	2,500	1,700
toluene	ug/l	800	880	200	623	1,200
chlorobenzene	ug/l	1,700	1,200	180	1,027	760
methanol (C)	ug/l	30,000	60,000	60,000	50,000	10,000
SEMI-VOLATILE ORGANIC COMPOUNDS						
2-chlorophenol	ug/l	<40	<20	<20	27	<20
PRIORITY POLLUTANT METALS						
zinc	mg/kg	8	54	35	30	10
OTHER ANALYSES						
BOD_5	mg/l BOD	123	84	82	96	97
COD	mg/l	I	I	I	I	I
TOC	mg/l	65	57	52	58	58
oil & grease	mg/l	40	19	NA	30	40
pH	pH units	6.0	6.0	6.0	6.0	6.0

B = Analyte found in blank as well as in sample. Value has not been corrected for the blank contamination but the concentration of the analytes found in the lab blanks are given below.
13138; acetone in lab blank = 8.0 ug/l
13141; acetone in lab blank = 9.8 ug/l

(C) = Evaluated by gas chromatograph/flame ionization detector.
I = Interference - All samples reduce $K_2Cr_2O_7$ prior to titration.
NA = Not analyzed.
* = Units are ug/kg
Analyte concentrations below detection limit were considered at detection limit for calculation of averages.

TABLE 51. FACILITY I INFLUENT TO AERATION GRAB SAMPLES -- DAY 3

Pollutant	Units	Day 3-Grab#1 Influent to Aeration 13165	Day 3-Grab#2 Influent to Aeration 13166	Day 3-Grab#3 Influent to Aeration 13167	Day 3-Grab Average Influent to Aeration	Day 3-24 Hr. Comp Influent to Aeration 13168
VOLATILE ORGANIC COMPOUNDS						
methylene chloride	ug/l	69 B	48	60 B	59	65
acetone (2-propanone)	ug/l	780 B	720	810 B	770	1,200
2-butanone	ug/l	88	<50	<120	86	<120
4-methyl-2-pentanone	ug/l	2,100	2,300	5,400	3,267	6,000
toluene	ug/l	2,100	5,000	2,600	3,233	2,600
chlorobenzene	ug/l	710	320	380	470	260
methanol (C)	ug/l	<10,000	40,000	<10,000	20,000	30,000
SEMI-VOLATILE ORGANIC COMPOUNDS						
2-chlorophenol	ug/l	<20	<20	<20	<20	<20
PRIORITY POLLUTANT METALS						
zinc	mg/kg	50	50	130	77	25
OTHER ANALYSES						
BOD_5	mg/l BOD	104	172	119	132	122
COD	mg/l	I	I	I	I	I
TOC	mg/l	340	78	73	164	70
oil & grease	mg/l	6	13	NA	9	3
pH	pH units	5.5	6.0	6.0	5.8	5.5

A = Sample had both liquid and semi-solid phases. TOC is calculated as the weighted average of the two phases and is expressed as ppm on a weight basis.

B = Analyte found in blank as well as in sample. Value has not been corrected for the blank contamination but the concentration of the analytes found in the lab blanks are given below.
13165,13167: methylene chloride in lab blank = 1.8 ug/l
acetone in lab blank = 11 ug/l

(C) = Evaluated by gas chromatograph/flame ionization detector.
I = Interference - All samples reduce $K_2Cr_2O_7$ prior to titration.
NA = Not analyzed.
* = Units are ug/kg
Analyte concentrations below detection limit were considered at detection limit for calculation of averages.

TABLE 52. FACILITY I AFI GRAB SAMPLES -- DAY 1

Pollutant	Units	Day 1-Grab#1 AFI Waste 13122	Day 1-Grab#2 AFI Waste 13123	Day 1-Grab#3 AFI Waste 13124	Day 1-Grab Average AFI Waste	Day 1 24 Hr Comp AFI Waste 13125
VOLATILE ORGANIC COMPOUNDS						
methylene chloride	ug/l	<5	<5 B	<5	<5	<5
acetone (2-propanone)	ug/l	150	105	200 B	152	180
2-butanone	ug/l	14	<10	15	13	<10
4-methyl-2-pentanone	ug/l	<10	<10	<10	<10	<10
toluene	ug/l	<5	<5	<5	<5	<5
chlorobenzene	ug/l	<5	<5	<5	<5	<5
methanol (C)	ug/l	<10,000	<10,000	<10,000	<10,000	10,000
SEMI-VOLATILE ORGANIC COMPOUNDS						
2-chlorophenol	ug/l	<20	<20	<20	<20	<20
PRIORITY POLLUTANT METALS						
zinc	mg/kg	8	60	20	29	35
OTHER ANALYSES						
BOD_5	mg/l BOD	1,060	1,340	1,590	1,330	914
COD	mg/l	I	I	I	I	I
TOC	mg/l	160	940	1,400	833	640
oil & grease	mg/l	84	107	1,480	558	569
pH	pH units	8.0	7.0	8.0	7.5	6.5

B = Analyte found in blank as well as in sample. Value has not been corrected for the blank contamination but the concentration of the analytes found in the lab blanks are given below.
13123; methylene chloride in lab blank = 2.1 ug/l
13124; acetone in lab blank = 14 ug/l

(C) = Evaluated by gas chromatograph/flame ionization detector.
I = Interference - All samples reduce $K_2Cr_2O_7$ prior to titration.
NA = Not analyzed.
* = Units are ug/kg
Analyte concentrations below detection limit were considered at detection limit for calculation of averages.

TABLE 53. FACILITY I AFI GRAB SAMPLES -- DAY 2

Pollutant	Units	Day 2-Grab#1 AFI Waste 13149	Day 2-Grab#2 AFI Waste 13150	Day 2-Grab#3 AFI Waste 13151	Day 2-Grab Average AFI Waste	Day 2 24 Hr Comp AFI Waste 13152
VOLATILE ORGANIC COMPOUNDS						
methylene chloride	ug/l	7.0	6.0	10	7.7	<62 B
acetone (2-propanone)	ug/l	180 B	45 B	35 B	87	<120 B
2-butanone	ug/l	<10	<10	<10	<10	<120
4-methyl-2-pentanone	ug/l	<10	<10	<10	<10	<120
toluene	ug/l	<5	<5	<5	<5	<62
chlorobenzene	ug/l	<5	<5	<5	<5	<62
methanol (C)	ug/l	<10,000	<10,000	<10,000	<10,000	<10,000
SEMI-VOLATILE ORGANIC COMPOUNDS						
2-chlorophenol	ug/l	<20	<40	<20	<27	<20
PRIORITY POLLUTANT METALS						
zinc	mg/kg	NA	5	6	5	6
OTHER ANALYSES						
BOD_5	mg/l BOD	735	917	833	828	791
COD	mg/l	I	I	I	I	I
TOC	mg/l	760	770	980	837	550
oil & grease	mg/l	715	579	248	514	235
pH	pH units	9.0	9.0	8.5	8.8	8.5

B = Analyte found in blank as well as in sample. Value has not been corrected for the blank contamination but the concentration of the analytes found in the lab blanks are given below.
13149; acetone in lab blank = 9.8 ug/l
13150, 13151; acetone in lab blank = 8.0 ug/l
13152; methylene chloride in lab blank = 1.8 ug/l
acetone in lab blank = 11 ug/l

(C) = Evaluated by gas chromatograph/flame ionization detector.
I = Interference - All samples reduce $K_2Cr_2O_7$ prior to titration.
NA = Not analyzed.
* = Units are ug/kg
Analyte concentrations below detection limit were considered at detection limit for calculation of averages.

TABLE 54. FACILITY I AFI GRAB SAMPLES -- DAY 3

Pollutant	Units	Day 3-Grab#1 AFI Waste 13182	Day 3-Grab#2 AFI Waste 13183	Day 3-Grab#3 AFI Waste 13184	Day 3-Grab Grab Average	Day 3 24 Hr Comp AFI Waste 13185
VOLATILE ORGANIC COMPOUNDS						
methylene chloride	ug/l	190	69	<62	<107	<62
acetone (2-propanone)	ug/l	<120	<10	170	<100	140
2-butanone	ug/l	<120	<10	<120	<83	<62
4-methyl-2-pentanone	ug/l	<120	<10	<120	<83	<120
toluene	ug/l	<62	<5	<62	<43	<62
chlorobenzene	ug/l	<62	<5	<62	<43	<62
methanol (C)	ug/l	<10,000	<10,000	<10,000	<10,000	<10,000
SEMI-VOLATILE ORGANIC COMPOUNDS						
2-chlorophenol	ug/l	<20	<20	<20	<20	<20
PRIORITY POLLUTANT METALS						
zinc	mg/kg	<0.2	<0.2	3	<1.1	10
OTHER ANALYSES						
BOD_5	mg/l BOD	1,120	3,550	1420	2,030	980
COD	mg/l	I	I	I	I	I
TOC	mg/l	850	2,100	1,400	1,450	620
oil & grease	mg/l	480	280	997	562	135
pH	pH units	8.5	8.5	9.0	8.7	8.0

(C) = Evaluated by gas chromatograph/flame ionization detector.
I = Interference - All samples reduce $K_2Cr_2O_7$ prior to titration.
NA = Not analyzed.
* = Units are ug/kg
Analyte concentrations below detection limit were considered at detection limit for calculation of averages.

TABLE 55. FACILITY I SECONDARY EFFLUENT GRAB SAMPLES -- DAY 1

Pollutant	Units	Day 1-Grab#1 Secondary Effluent 13115	Day 1-Grab#2 Secondary Effluent 13116	Day 1-Grab#3 Secondary Effluent 13117	Day 1-Grab Average Secondary Effluent	Day 1-24 Hr. Comp Secondary Effluent 13118
VOLATILE ORGANIC COMPOUNDS						
methylene chloride	ug/l	79 B	9	<5 B	<31	7
acetone (2-propanone)	ug/l	<10	13	31 B	<18	27 B
2-butanone	ug/l	<10	<10	<10	<10	<10
4-methyl-2-pentanone	ug/l	<10	<10	<10	<10	<10
toluene	ug/l	6.7	<5	<5	<6	<5
chlorobenzene	ug/l	<5	<5	<5	<5	<5
methanol (C)	ug/l	<10,000	<10,000	<10,000	<10,000	<10,000
SEMI-VOLATILE ORGANIC COMPOUNDS						
2-chlorophenol	ug/l	<20	<20	<20	<20	<20
PRIORITY POLLUTANT METALS						
zinc	mg/kg	6	7	9	7	30
OTHER ANALYSES						
BOD_5	mg/l BOD	<6	<6	<6	<6	<6
COD	mg/l	I	I	I	I	I
TOC	mg/l	12	16	14	14	13
oil & grease	mg/l	0.7	25	69	32	62
pH	pH units	6.5	6.5	6.5	6.5	6.0

B = Analyte found in blank as well as in sample. Value has not been corrected for the blank contamination but the concentration of the analytes found in the lab blanks are given below.
13117; methylene chloride in lab blank = 3.0 ug/l
acetone in lab blank = 14 ug/l
benzene in lab blank = 1.2 ug/l
13118; acetone in lab blank = 14 ug/l

(C) = Evaluated by gas chromatograph/flame ionization detector.
I = Interference - All samples reduce $K_2Cr_2O_7$ prior to titration.
NA = Not analyzed.
* = Units are ug/kg
Analyte concentrations below detection limit were considered at detection limit for calculation of averages.

TABLE 56. FACILITY I SECONDARY EFFLUENT GRAB SAMPLES -- DAY 2

Pollutant	Units	Day 2-Grab#1 Secondary Effluent 13142	Day 2-Grab#2 Secondary Effluent 13143	Day2-Grab#3 Secondary Effluent 13144	Day 2-Grab Average Secondary Effluent	Day 2-24 Hr. Comp Secondary Effluent 13145
VOLATILE ORGANIC COMPOUNDS						
methylene chloride	ug/1	5.0	5.0	5.0	5.0	<5
acetone (2-propanone)	ug/1	<10	<10	<10	<10	<10
2-butanone	ug/1	<10	<10	<10	<10	<10
4-methyl-2-pentanone	ug/1	<10	<10	<10	<10	<10
toluene	ug/1	<5	<5	<5	<5	<5
chlorobenzene	ug/1	<5	<5	<5	<5	<5
methanol (C)	ug/1	<10,000	<10,000	<10,000	<10,000	<10,000
SEMI-VOLATILE ORGANIC COMPOUNDS						
2-chlorophenol	ug/1	<40	<20	<20	27	<20
PRIORITY POLLUTANT METALS						
zinc	mg/kg	6	5	100	37	7
OTHER ANALYSES						
BOD_5	mg/1 BOD	<6	<6	<6	<6	<12
COD	mg/1	I	I	I	I	I
TOC	mg/1	13	15	13	14	17
oil & grease	mg/1	84	21	40	48	83
pH	pH units	6.5	6.5	6.5	6.5	6.5

(C) = Evaluated by gas chromatograph/flame ionization detector.
I = Interference - All samples reduce $K_2Cr_2O_7$ prior to titration.
NA = Not analyzed.
* = Units are ug/kg
Analyte concentrations below detection limit were considered at detection limit for calculation of averages.

TABLE 57. FACILITY I SECONDARY EFFLUENT GRAB SAMPLES -- DAY 3

Pollutant	Units	Day 3-Grab#1 Secondary Effluent 13169	Day 3-Grab#2 Secondary Effluent 13170	Day 3-Grab#3 Secondary Effluent 13171	Day 3-Grab Average Secondary Effluent	Day 3-24 Hr. Comp Secondary Effluent 13172
VOLATILE ORGANIC COMPOUNDS						
methylene chloride	ug/l	<62 B	<62 B	*62 B	<62	5.0 B
acetone (2-propanone)	ug/l	<120 B	<120 B	<120 B	<120	<120
2-butanone	ug/l	<120	<120	<120	<120	<120
4-methyl-2-pentanone	ug/l	<120	<120	<120	<120	<120
toluene	ug/l	<62	<62	<62	<62	<62
chlorobenzene	ug/l	<62	<62	<62	<62	<62
methanol (C)	ug/l	<10,000	<10,000	<10,000	<10,000	<10,000
SEMI-VOLATILE ORGANIC COMPOUNDS						
2-chlorophenol	ug/l	<20	<20	<10	<20	20
PRIORITY POLLUTANT METALS						
zinc	mg/kg	9	8	6	8	10
OTHER ANALYSES						
BOD_5	mg/l BOD	<12	<20	<20	17	<6
COD	mg/l	I	I	I	I	I
TOC	mg/l	13	12	12	12	12
oil & grease	mg/l	91	55	112	86	65
pH	pH units	6.5	6.5	6.0	6.3	6.0

B = Analyte found in blank as well as in sample. Value has not been corrected for the blank contamination but the concentration of the analytes found in the lab blanks are given below.
13169, 13170, 13171, 13172; methylene chloride in lab blank = 1.8 ug/l
acetone in lab blank = 11 ug/l

(C) = Evaluated by gas chromatograph/flame ionization detector.
I = Interference - All samples reduce $K_2Cr_2O_7$ prior to titration.
NA = Not analyzed.
* = Units are ug/kg
Analyte concentrations below detection limit were considered at detection limit for calculation of averages.

value of 131 µg/l. 4 methyl-2 pentanone concentrations ranged from <10 µg/l to 5,400 µg/l with a mean of 2,557 µg/l. Toluene concentrations were found in the range of <5 to 5,000 µg/l with a mean concentration of 1,460 µg/l. Chlorobenzene was also present in the concentration range from <5 to 1,700 µg/l and averaged 540 µg/l. Methanol was found in the greatest concentration ranging from <10,000 to 120,000 µg/l with a mean of 56,667 µg/l.

Zinc was detected in concentrations ranging from 6 to 130 mg/kg and averaged 38.7 mg/kg.

The pH of the discrete grab samples was relatively consistent and ranged from 5.5 to 6.0. Increased methanol concentrations may have contributed substantially to BOD_5 and TOC.

Analyte concentrations in daily 24-hour composite samples were consistent with daily averages of grab samples. These results are presented in Tables 48-51.

AFI Waste --

Data regarding the AFI waste stream is presented in Tables 48 and 52-54. Most volatile and semi-volatile organic compounds were found at or below detection limits in the nine grab samples collected. The only measurable organic compounds included methylene chloride, acetone (2 propanone) and 2 butanone. Methylene chloride concentrations ranged from <5 to 190 µg/l with a mean value of 40 µg/l. Acetone was measured above the detection limit in seven of the nine grab samples and ranged from <10 to 200 µg/l with an average concentration of <113 µg/l. 2 butanone concentration ranged from <10 to <120 µg/l with a mean concentration of <35 µg/l.

Zinc concentrations varied from <0.2 to 60 mg/kg with a mean value of <13 mg/kg. Lowest zinc concentrations were measured on day 3 of the sampling episode.

Biochemical oxygen demand (BOD_5), TOC, and oil and grease were relatively high and consistent with the organic nature of the waste. BOD_5 concentrations ranged from 735 to 3,550 mg/l with an average value of 1,396 mg/l. TOC concentrations ranged from 160 to 2,100 mg/l with a mean of 1,040 mg/l. Oil and grease concentrations varied from 87 to 1,480 mg/l with an average of 443 mg/l.

pH of this waste stream ranged from 7.0 to 9.0 with an average of 8.3.

Analyte concentrations of 24-hour comp samples collected are presented in Tables 52-54 for Day 1, Day 2 and Day 3 respectively and are consistent with the daily average concentrations of grab samples.

Secondary Effluent --

Data regarding secondary effluent sample analyte concentrations are presented in Tables 48 and Tables 55-57 Methylene chloride and acetone were the only two organic compounds found above analyte detection limits. Methylene chloride concentrations ranged from <5 to 79 µg/l with a mean value of <31 µg/l. Acetone concentrations ranged from <10 to 31 µg/l with an average concentration of <18 µg/l. BOD_5 and TOC concentrations were relatively low indicating efficient removal. Oil and grease values were variable ranging from <0.7 mg/l to 112 mg/l. pH was 6.5 for all samples. Zinc was measured in concentrations ranging from 5 to 100 mg/kg with an average value of 17 mg/kg.

24-hour comp results were consistent with daily grab sample averages as presented in Tables 55-57.

Waste Activated Sludge --

Nine grab samples of waste activated sludge were collected during the sampling episode. The calculated average analyte concentrations of these samples are presented in Table 48. Methylene chloride was detected in all samples with a concentration range of 21 µg/l to 2,400 µg/l. Acetone was measured in the concentration range of <5 to 220 µg/l. As expected, zinc concentrations were relatively high ranging from 770 to 1,600 mg/kg. The pH was consistent at 6.5.

EVALUATION OF EFFECTIVENESS

To determine removal efficiencies, analyte concentrations of influent to aeration and AFI waste were compared with secondary effluent concentrations of the 24 hour daily composite samples Tables 49-57. In addition the removal efficiencies for the average concentration state the nine discrete grab samples (Table 48) were calculated.

Volatile organic compound removal efficiencies were generally >90% in 24 hr composites, although methylene chloride and 2 butanone removal efficiencies were decreased to approximately 60% based on the mean of the nine discrete samples. Methanol removal ranged from 64-92%. This may be attributed to the relatively high detection limit of <10,000 µg/l. TOC and BOD_5 removal were greater than 85 and 94% respectively. Oil and grease removal was relatively low (<14%), zinc removal efficiencies ranged from 38-85%.

RESIDUE MANAGEMENT

The primary and secondary sludge is filtered using vacuum filtration or a plate and frame filter press and placed in a RCRA surface impoundment adjacent to Facility I. The secondary effluent is discharged into an adjacent river tributary.

SECTION 12

CARBON ADSORPTION

Carbon adsorption was the final unit operation in aqueous waste treatment trains at two facilities, B and C. In both cases, granular activated carbon was used to remove dissolved organics after upstream processes removed heavy metals and suspended solids.

THEORY

Activated carbon is used for removal of dissolved organic compounds from aqueous wastes. Organic molecules are adsorbed, i.e., attracted to and held on the surface of the activated carbon by physical and/or chemical forces. Adsorption is caused by intermolecular forces, primarily Van der Waal's forces, between the pores and surfaces of the solid adsorbent and organic solutes. When the attractive forces between the solute and adsorbent exceed those forces between the solute and solvent, the solute is adsorbed onto the surface and pores of the adsorbent. Granular activated carbon (GAC) adsorption is a well established and widely used technology in water treatment as well as in the chemical process industries.

The technical evaluation of activated carbons is best served by determining the adsorption rate and capacity of the different carbons. Activated carbons are made from several different bases including shells, wood, petroleum derivatives, peat and lignite and bituminous coals. All of these carbons demonstrate different physical characteristics and adsorbent capacities. The effectiveness of activated carbon for adsorption is related to its large surface area. Surface area of typical activated carbons can range from 500 to 1400 square meters per gram of carbon.

The adsorption isotherm is the relationship, at a given temperature and other specified conditions, between the amount of substance adsorbed and its concentration in the surrounding solution. An isotherm consists of a curve plotted with residual concentration of organics in the wastewater on the abscissa, and the quantity or organics adsorbed per gram of carbon as the ordinate. A reading taken at any point on the isotherm gives the quantity of organics adsorbed per unit weight of carbon, which is the carbon adsorptive capacity for a particular organic concentration and wastewater temperature. In a very dilute solution, such as wastewater, a logarithmic isotherm plotting usually yields a straight line. In this connection, a useful formula is the Freundlich equation which relates the amount of impurity in the solution to that adsorbed as follows:

$$x/m = kC^{1/n}$$

where:

x = amount of TOC or other constituent adsorbed

m = weight of carbon

k & n are constants

C = unadsorbed core of TOC or other constituent left in solution

in logarithmic form:

$$\log x/m = \log k + Yn \log C$$

in which $1/n$ represents the slope of the straight line isotherm.

By performing bench scale tests or by obtaining isotherm data from previous studies, it can be determined whether or not a particular degree of organic removal can be effected by adsorption alone. It will also show the approximate adsorptive capacity of the carbon for the application.

Figure 22 illustrates a typical fixed-bed adsorption system using a single carbon column with downflow of the liquid. During adsorption, the wastewater flows through an inlet distributer at the top of the column, flows downward through the bed and leaves through the underdrain system. Typical hydraulic flow rates vary from 2 to 5 gpm/ft^2. A breakthrough curve (Figure 23) plots the solute concentration in the effluent against the volume of effluent against the volume of effluent treated. The carbon bed is backwashed, when a certain headloss is exceeded, due to accumulated suspended solids. Once the contaminant of interest exceeds the specified effluent concentration, the carbon bed must be regenerated using a furnace, which is usually a multiple hearth type furnace.

As carbon becomes saturated, the zone of adsorption (the length of the column in which adsorption occurs) moves downward with only a gradual increase in the concentration of organics in the carbon effluent until this zone reaches the discharge point in the column. At this time the organic concentration increases until it is equal to the influent concentration. Adsorption is most effective when the organic molecules to be removed have high molecular weight, high boiling points, low aqueous solubility, and low polarity.

FACILITY B

Process Description

Unit processes for treatment of aqueous wastes at Facility B include storage, blending, pH adjustment, chemical reduction, lime precipitation of heavy metals, filtration, neutralization and carbon adsorption. Influent cyanide wastes are subjected to alkaline chlorination pretreatment prior to

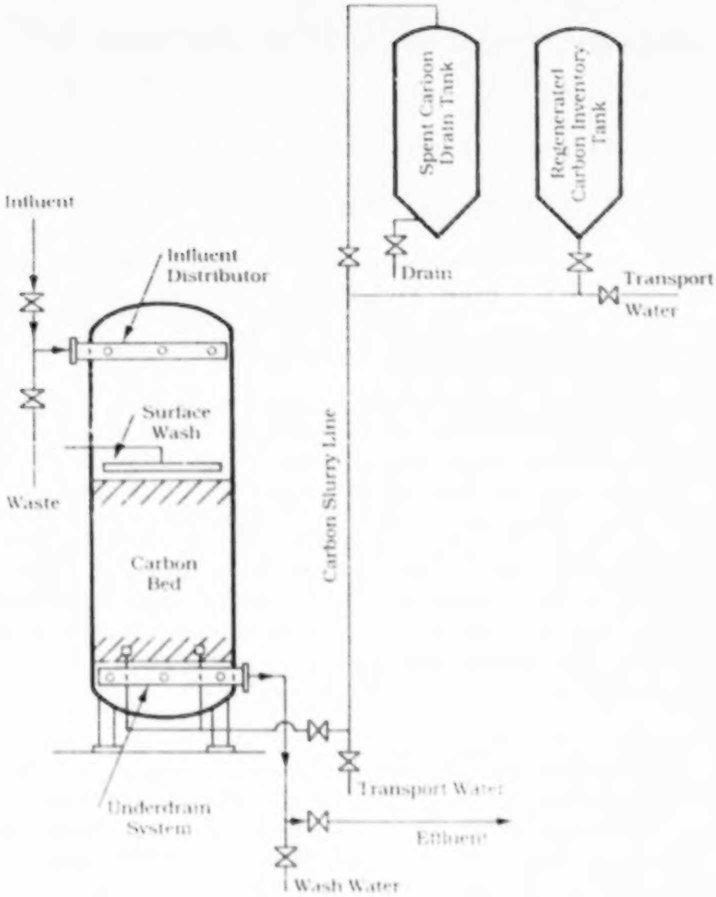
Influent
Influent Distributor
Surface Wash
Waste
Carbon Bed
Underdrain System
Carbon Slurry Line
Spent Carbon Drain Tank
Regenerated Carbon Inventory Tank
Drain
Transport Water
Transport Water
Effluent
Wash Water

Figure 22. Schematic of Fixed Bed Adsorption System

(Reynolds, Tom D., Unit Operations and Processes in Environmental Engineering, Wadsworth, Inc., 1982, p. 192)

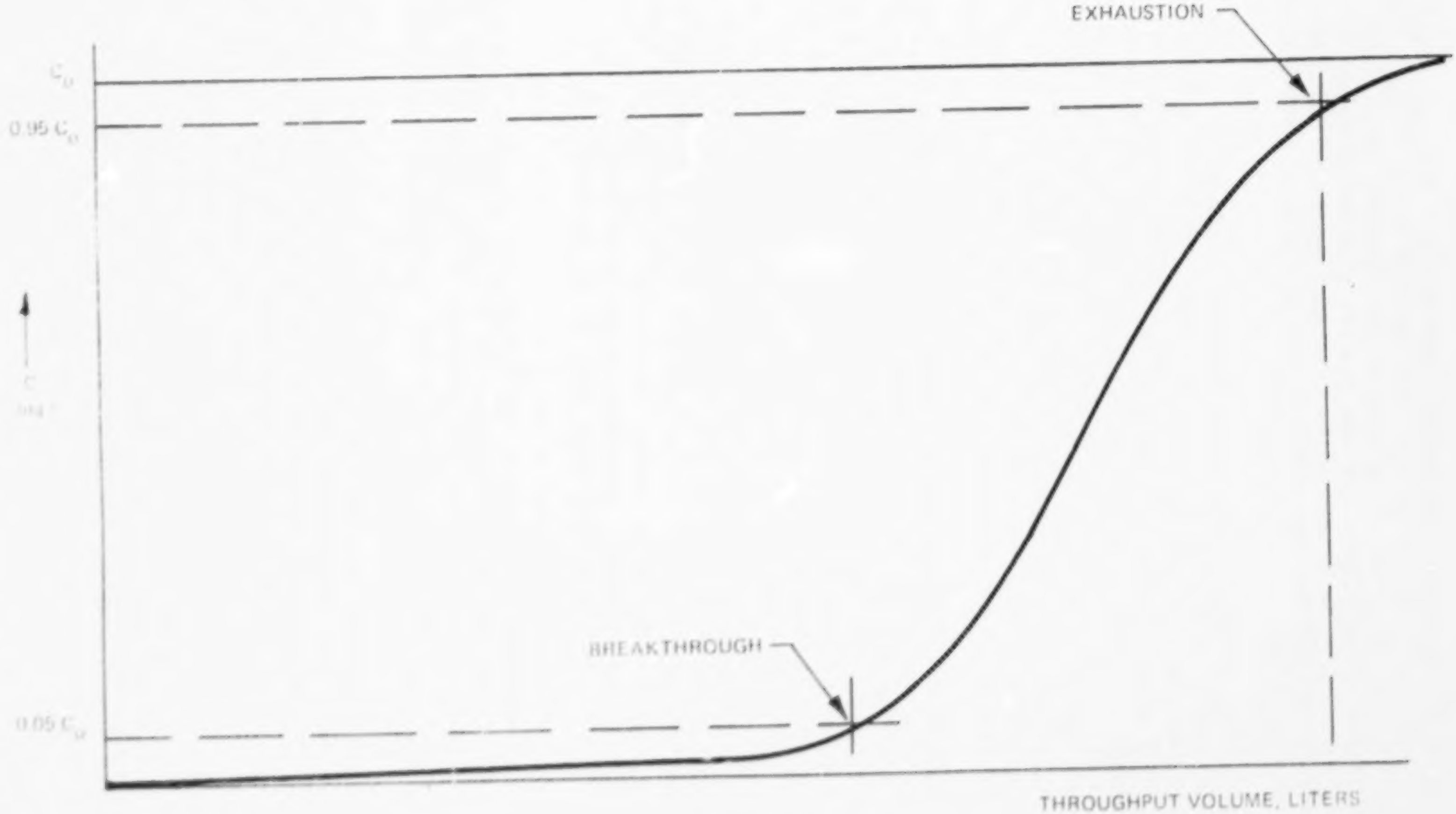

FIGURE 23. TYPICAL CARBON BREAKTHROUGH CURVE

being pumped to the aqueous waste treatment system. Ordinarily this waste constitutes about 25 percent of the total aqueous waste volume. However, at the time of sampling, about 75 percent of the incoming waste to the aqueous waste treatment was effluent from the cyanide alkaline chlorination pretreatment process. The aqueous waste treatment system is designed to handle about 150,000 gallons per day (GPD), but presently is operating at only 30,000 to 40,000 GPD.

Noncyanide, dilute aqueous waste received at the plant is pumped to one of two wastewater receiving/equalization tanks. These are referenced as tanks T-5 and T-45 in Figure 24. This wastewater may contain total organic carbon (TOC) concentrations as high as 10,000 mg/l, in addition to a high metal content. The effluent from the alkaline chlorination process is also pumped to the receiving/equalization tanks. The equalization capacity of each receiving tank, nominally 50,000 gallons per tank, is diminished by a bottom layer of settled solids and a top layer of light organics. Management indicated that conical bottom tanks and scum removal equipment to maximize tank usage would be incorporated in future receiving tank designs.

Blended wastewater from the equalization tanks is pumped to one of two 13,900 gallon capacity pH reduction tanks (T-55 and T-56). The pH of the waste is adjusted to 2 with dilute (approximately 20 percent) mixed waste acid (sulfuric, nitric, hydrochloric, or chromic acid) from tank T-58 and/or virginhydrochloric acid from tank T-67. The pH adjustment serves to break up certain metal-organic chelate complexes, facilitating metals precipitation in the next unit operation. In addition, this operation also serves to introduce the acid wastes into the aqueous waste treatment system for dilution and subsequent metal removal and neutralization. When sulfuric acid is added to the waste, calcium sulfate is formed, which enhances the downstream filter press operation but also increases the quantity of sludge generated. If chromic acid is added to the waste (tank T-53), then bisulfite (tank T-72) is also added to reduce hexavalent chromium to trivalent form. The waste is agitated in the pH reduction tank for approximately two to three hours.

After pH adjustment to about 2, the wastewater is pumped to a 6,000 gallon steel reactor vessel (R-2) where lime is added. This raises the pH to approximately 11, resulting in the formation of insoluble metal hydroxides. Lime is added to the reactor prior to adding the acidified waste to protect the steel tank from corrosion.

Following lime addition, the wastewater (now a slurry of metal precipitates in water) is transferred from the precipitation tank to one of three 17,000 gallon capacity aqueous waste slurry surge tanks (T62, T63 and T64). The slurry is agitated in the surge tanks and is pumped to a plate and frame filter press for dewatering. This press produces a sludge filter cake with a solids content of about 45 percent. The press currently uses polypropylene fabric filter material and no chemical conditioning. The dewatered sludge, typically containing cadmium, nickel, and/or lead, is trucked off-site for land disposal. The filtrate from the press is neutralized to a pH of 7.5 with reagent grade hydrochloric acid in a 5000

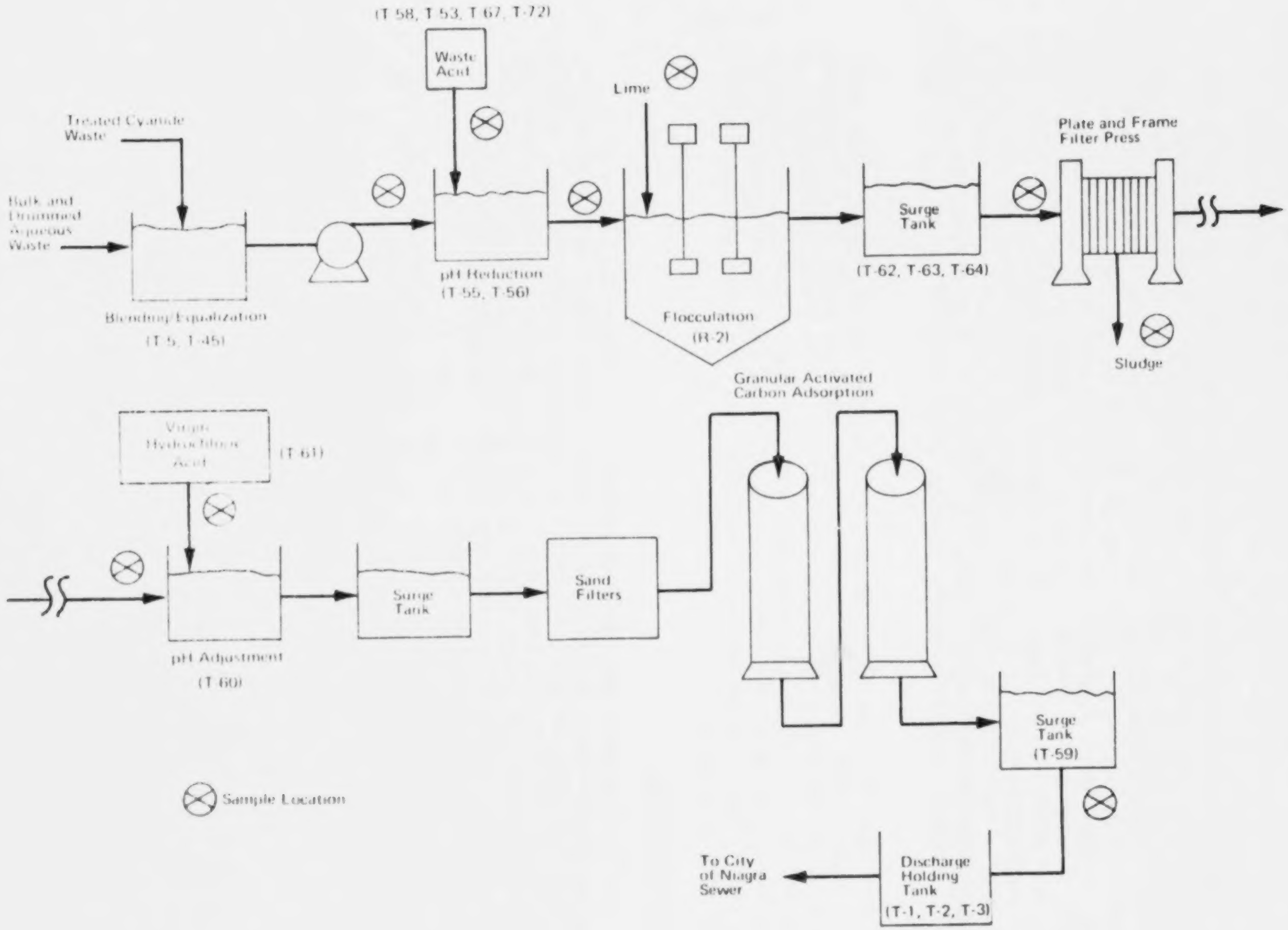
(T-58, T-53, T-67, T-72)
Waste Acid
Treated Cyanide Waste
Lime
Plate and Frame Filter Press
Bulk and Drummed Aqueous Waste
Surge Tank
(T-62, T-63, T-64)
pH Reduction
(T-55, T-56)
Blending/Equalization
(T-5, T-45)
Flocculation
(R-2)
Sludge
Granular Activated Carbon Adsorption
Virgin Hydrochloric Acid
(T-61)
Surge Tank
Sand Filters
pH Adjustment
(T-60)
Surge Tank
(T-59)
Sample Location
To City of Niagra Sewer
Discharge Holding Tank
(T-1, T-2, T-3)

FIGURE 21. FACILITY B FLOW DIAGRAM/SAMPLE POINTS FOR AQUEOUS WASTE TREATMENT SYSTEM.

gallon capacity pH adjustment tank (T60) and then passed through two multimedia filters in series, followed by two beds of granular activated carbon (GAC) in series before discharge to the local municipal sewer system.

The carbon adsorbers are leased from Calgon, who is responsible for carbon removal and regeneration. Adsorption on GAC is the principal mechanism for removal of organics in Facility B's aqueous waste treatment process. Phenol breakthrough (defined as a significant increase in effluent concentration, generally about 5 to 10 percent of the influent concentration) is used as an indicator for carbon exhaustion, although TOC gradually breaks through before phenol (typically after about 14 days of operation). From that point on, effluent TOC is high and fairly constant at 1000 - 2000 mg/l (representing 80 to 90 percent removal of the influent maximum 10,000 mg/l TOC). Facility B's sewer discharge permit allows a maximum daily TOC concentration of 1500 mg/l. Phenol is limited to 6.16 mg/l by the discharge permit. Phenol typically breaks through after about 20 to 25 days of operation.

Effluent from the GAC adsorbers is transferred to a small surge tank (T59), then pumped to one of three effluent storage tanks (T1, T2 and T3), each with a capacity of about 105,000 gallons. The three effluent storage tanks can hold up to about three days accumulation of treated wastewater prior to discharge to the sewer system. If discharge requirements are not met, the effluent is pumped back to the head of the treatment system for additional treatment.

Sampling Program

During the three day sampling episode at Facility B samples of the filter press filtrate (granulated activated carbon (GAC) influent) and the granular activated carbon effluent were composited once each day. The sampling point locations are shown in Figure 22. The samples of the influent to the GAC column were taken prior to the pH adjustment tank and sand filtration units. Table 58 presents the sample locations and procedures for the granular activated carbon system.

TABLE 58. FACILITY B SAMPLING LOCATIONS AND PROCEDURES

Sample Description	Sample Location	Sampling Procedure
Filter Press Filtrate (GAC Influent)	Samples were taken prior to the neutralization in the pH adjustment tank (T60)	Grab samples were collected and composited each day during a 10 hour interval. Typically this interval extended from 0800 hours to 1800 hours.
GAC Effluent	Samples were taken from the effluent surge tank (T-59)	Samples were procured by an ISCO automatic sampler (500 ml every 20 minutes) over a 10 hour interval for each of the three days.

Data

Influent Characterization --

Table 59 summarizes analytical data for Facility B's carbon adsorption process. The filtrate or influent to the GAC adsorbers had a total organic concentration ranging from 2,400 mg/l to 5,000 mg/l and averaging 3,300 mg/l during the three day sampling program. Total solids concentration ranged from 80,000 to 90,000 mg/l and averaged 87,000 mg/l. Of the total solids, greater than 80% was dissolved. Individual organic compounds measured in all three GAC influent samples were methylene chloride, acetone, toluene, 2-nitrophenol, 4-nitrophenol, and phenol. Significant concentrations of six heavy metals-cadmium, chromium, copper, lead, nickel and zinc- were also measured in the filter press filtrate. Copper and zinc were the major metals with average concentrations of 237 mg/l and 29 mg/l, respectively.

Effluent Characterization--

Facility B's GAC effluent had an average TOC concentration of 1,367 mg/l. Total solids concentration ranged from 60,000 mg/l to 87,000 mg/l, with an average value of 74,300 mg/l of which greater than 98% was dissolved. Acetone was reported in GAC effluent samples from each of the three sampling days. Acetone concentration ranged from 55,000)g/l to 160,000)g/l. No other specific organic compounds were reported in the GAC effluent samples from days 1 and 2. However, the day 3 GAC effluent sample contained 30,000)g/l methylene chloride and 1,600 mg/l trichloroethylene as well as 55,000)g/l acetone, GAC effluent metals concentrations were low. Nickel, with an average concentration of 2.0 mg/l, was present in highest concentrations.

TABLE 59. FACILITY B CARBON ADSORPTION DATA

Parameter	Units	Day 1 Filtrate	Day 1 GAC Effluent	Day 2 Filtrate	Day 2 GAC Effluent	Day 3 Filtrate	Day 3 GAC Effluent
VOC							
Methylene chloride	µg/L	220,000	<1,250	43,000	<2,500	130,000	30,000
Acetone	µg/L	68,000	140,000	61,000	160,000	69,000	55,000
2-butanone (MEK)	µg/L	<5,000	<2,500	6,600	<5,000	7,000	<5,000
1,1,1-trichloroethane	µg/L	ND	ND	2,500	<1,000	1,600	1,600
Trichloroethene	µg/L	4,200	<500	1,400	<1,000	<1,000	<1,000
Toluene	µg/L	13,000	<500	7,700	<1,000	3,900	<1,000
Total xylenes	µg/L	<1,000	<500	8,000	<1,000	<1,000	<1,000
Acid Extractables							
P-chloro-m-cresol	µg/L	5,600	<100	1,600	<100	<500	<100
2-nitrophenol	µg/L	1,200	<100	1,600	<100	3,000	<100
4-nitrophenol	µg/L	1,100	<100	2,300	<100	1,400	<100
2,4-dinitrophenol	µg/L	120	<100	<400	<100	ND	ND
Phenol	µg/L	19,000	<100	4,200	<100	5,100	<100
Base/Neutral Extractables							
N-nitrosodimethylamine	µg/L	100	<100	ND	ND	ND	ND
Metals							
Cadmium	mg/L	1.2	0.15	7.9	0.51	50	1.4
Hex. chromium	mg/L	NS	<20	NS	<5	NS	<5
Total chromium	mg/L	2.1	0.091	1.9	0.12	0.068	0.091
Copper	mg/L	160	0.32	170	0.87	380	0.56
Lead	mg/L	1.4	0.56	0.82	0.74	0.67	0.63
Nickel	mg/L	2.2	2.2	2.3	2.0	22	1.8
Zinc	mg/L	24	1.4	16	1.6	46	0.70

TABLE 59. FACILITY B CARBON ADSORPTION DATA (cont.)

		Day 1		Day 2		Day 3	
Parameter	Units	Filtrate	GAC Effluent	Filtrate	GAC Effluent	Filtrate	GAC Effluent
Other Analyses							
Total Organic Carbon	mg/L	2,500	1,200	2,400	1,500	5,000	1,400
Total Solids	mg/L	80,000	60,000	90,000	87,000	90,000	76,000
Total Volatile Solids	mg/L	5,900	4,500	9,400	18,000	16,000	10,000
Total Suspended Solids	mg/L	400	980	540	820	1,300	850
Total Dissolved Solids	mg/L	73,000	59,000	90,000	86,000	89,000	75,000
Total Vol. Sus. Solids	mg/L	25	170	49	100	190	130
Total Vol. Dis. Solids	mg/L	5,900	4,300	9,000	18,000	16,000	10,000

ND - Not detected
NS - Not sampled

Evaluation of Effectiveness

Facility B's GAC adsorbers were operated in order to reduce wastewater TOC and phenol concentrations to below the sewer discharge permit limits of 1500 mg/l and 6.16 mg/l, respectively. These goals were achieved. Phenol concentration was reduced by greater than 97 percent, with phenol effluent concentrations consistently below 100 mg/l. TOC reduction ranged from 37.5% to 73%, with effluent TOC concentrations of 1200 to 1500 mg/l.

FACILITY C

Process Description

Unit operations for treatment of aqueous wastes at Facility C include blending, pH adjustment, hydroxide precipitation of heavy metals, sedimentation, filtration, multiple-effect evaporation and carbon adsorption. Figure 25 is a simple flow diagram for the entire aqueous waste treatment process.

Metal-containing and other aqueous wastes received at Facility C are discharged from tank trucks to a natural clay-lined 1.6 million gallon surface impoundment designated Pond 2. Any sludge present settles to the bottom of Pond 2. Aqueous waste from Pond 2 is then pumped to a 2.5 million gallon surface impoundment, designated Pond 6, for pretreatment. Wastewater from another surface impoundment, Pond 1, which holds oily waste is also pumped to Pond 6. In addition, rainwater runoff from three landfill cells (3,4,7) goes into Pond 6. When the level of wastewater in Pond 6 has reached some specified level (this occurs roughly once every two months), flow into this pond is stopped and a recirculating pump is started to pump wastewater through a baffled pretreatment tank located adjacent to Pond 6 and back into the pond. Treatment chemicals are added to this tank for mixing. First, waste acid is added to facilitate phase separation of organics. Then a polymer is added to help coagulate solids, and, finally, sodium hydroxide is added to effect precipitation of heavy metals as their hydroxides. Following reagent addition and mixing, the recirculation pump is shut off and Pond 6 is left undisturbed for a two-day period of quiescent settling. After sedimentation in Pond 6, the supernatant wastewater is pumped to Pond 8, a 7.4 million gallon holding basin. Water from the holding basin is continuously pumped through a sand/rock filter to the evaporator feed tank located inside the aqueous waste treatment building. The building houses a four-effect falling film evaporator used to concentrate hazardous solids while producing purified water.

A portion of the concentrated effluent from the evaporator is recycled and the remainder is pumped to Pond 6. A portion of the condensed overhead vapor is sent to the cooling tower as makeup water, the rest is pumped to a parallel set of sand filters. Effluent from the two sand filters is gravity fed to two open-top granular activated carbon (GAC) adsorber tanks in series. These serve to remove organics and low levels of certain inorganics. The effluent from the second GAC adsorber flows by gravity into a circular above-ground holding tank located outside the treatment building.

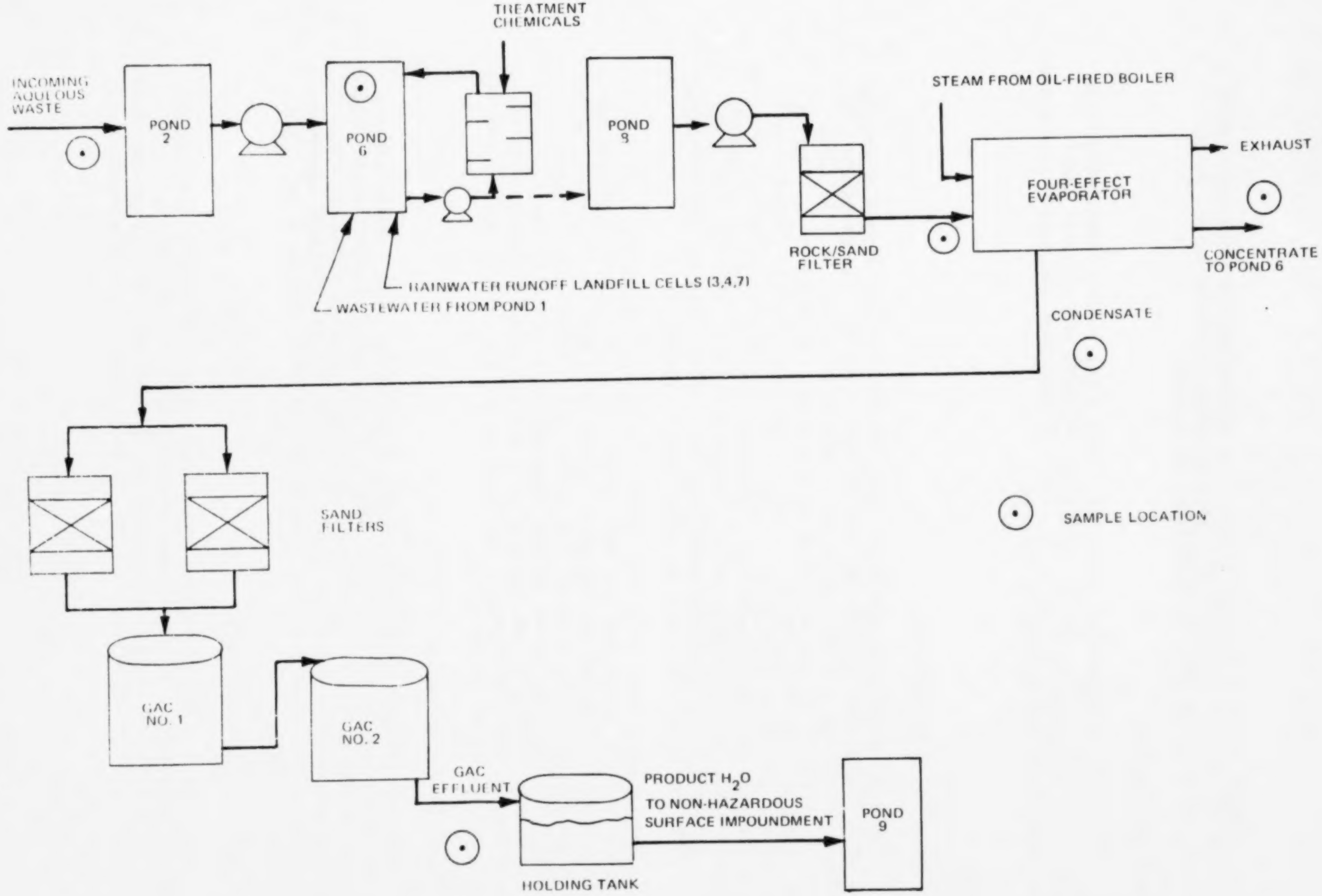
INCOMING AQUEOUS WASTE
POND 2
POND 6
TREATMENT CHEMICALS
POND 9
ROCK/SAND FILTER
STEAM FROM OIL-FIRED BOILER
FOUR-EFFECT EVAPORATOR
EXHAUST
CONCENTRATE TO POND 6
RAINWATER RUNOFF LANDFILL CELLS (3,4,7)
WASTEWATER FROM POND 1
CONDENSATE
SAND FILTERS
SAMPLE LOCATION
GAC NO. 1
GAC NO. 2
GAC EFFLUENT
HOLDING TANK
PRODUCT H_2O
TO NON-HAZARDOUS SURFACE IMPOUNDMENT
POND 9

FIGURE 25. FLOW DIAGRAM/SAMPLE POINTS FOR AQUEOUS WASTE TREATMENT AT FACILITY C.

This product water is currently sent to a nonhazardous surface impoundment on site for natural evaporation. When an NPDES discharge permit is obtained, this stream will be discharged to a creek.

Sampling Program

The sampling program at Facility C took place during three consecutive days in October, 1985. Carbon adsorption influent and effluent sample points are indicated on Figure 23. Table 60 describes the sample points and the procedures used to procure samples.

DATA

Influent Characterization--

Table 61 summarizes analytical data for Facility C's carbon adsorption influent (Evaporator Condensate) and effluent samples. The evaporator condensate stream was a clear, colorless liquid with a pH of 7. The total organic carbon (TOC) concentration of this stream averaged 56 mg/l during the three day sampling program. Total solids concentration ranged from 31 to 450 mg/l, with an average of 171 mg/l. The evaporator condensate samples were analyzed for thirteen metals - antimony, arsenic, cadmium, chromium, copper, iron, lead, mercury, nickel, selenium, silver, thallium and zinc. Only zinc was found above its detection limit, with measured concentrations of 0.0053 mg/l and 0.016 mg/l on days 1 and 3, respectively.

The organics data shows considerable variation from day to day, but samples from all three days had measurable levels of volatile and extractable organics. Acetone was the most significant organic contaminant, with a reported concentration range of 710 to 850 μg/l. Methyl ethyl ketone (1-butanone) was reported at greater than 200 μg/l in two of the three samples. Diethyl phthalate was measured at a concentration of 547 μg/l in the day 1 sample, and 2-nitrophenol was found at 222 μg/l and 75 μg/l in the day 1 and day 2 samples respectively.

Effluent Characterization--

The GAC effluent stream was crystal clear water, with a total organic carbon concentration of less than 1 mg/l. Total solids concentration ranged from 21 to 420 mg/l. No volatile or extractable organic compounds were reported above their detection limits in any of the GAC effluent samples. One metal, zinc, was reported at low concentration in the day 1 and day 3 effluent samples.

Evaluation of Effectiveness

Facility C's GAC adsorption process was very effective in reducing the organic content of a dilute aqueous waste stream. Total organic carbon (TOC) concentration removal efficiency was greater than 98%. Over the three day episode the primary compounds in the GAC influent such as acetone

TABLE 60. FACILITY C SAMPLE LOCATIONS AND PROCEDURES

Sample Reference	Description	Type of Sample	Time Interval
Evaporator Condensate	Samples were taken from the evaporator condensate sample tap. A beaker was used to take the sample aliquots to make up the composite.	Composite	Five grab aliquots of one gallon each were taken at 1.5 hour intervals during the evaporator operation and were composited to form a single sample each day. A single discrete grab sample for VOC analysis was collected each day.
GAC Effluent	Samples were taken from the GAC effluent sample tap located after the second GAC in series. A beaker was used to take the sample aliquots to make up the composite.	Composite	Same as above

TABLE 61. FACILITY C CARBON ADSORPTION DATA

Parameter	Units	Day 1 Evaporator Condensate	Day 1 GAC Effluent	Day 2 Evaporator Condensate	Day 2 GAC Effluent	Day 3 Evaporator Condensate	Day 3 GAC Effluent
VOC							
Methylene chloride	µg/L	34	<5	<5	<5	15	<5
Acetone	µg/L	710	<50	800	<50	850	<50
2-Butanone (MEK)	µg/L	<50	<10	360	<10	230	<10
1,1,1-trichloroethane	µg/L	<10	<2	<10	<10	34	<2
Trichloroethene	µg/L	<10	<2	<2	<2	7.5	<2
Benzene	µg/L	<10	<2	ND	ND	21	<2
4-methyl-2-pentanone	µg/L	<50	<10	26	<10	31	<10
Tetrachloroethene	µg/L	<10	<2	<2	<2	6.5	<2
Toluene	µg/L	<10	<2	9	<2	62	<2
Ethyl benzene	µg/L	<10	<2	<2	<2	5.5	<2
Total xylenes	µg/L	<10	<2	<2	<2	26	<2
Acid Extractables							
2,4-dichlorophenol	µg/L	ND	ND	37	<20	ND	ND
2-nitrophenol	µg/L	222	<20	75	<20	ND	ND
Base/Neutral Extractables							
Bis (2-chloroethyl) ether	µg/L	86	<20	<20	<20	ND	ND
Diethyl phthalate	µg/L	547	<20	<20	<20	<10	<10
isophorone	µg/L	ND	ND	21	<20	26	<10
Metals							
Zinc	mg/L	.0053	.027	<.005	<.005	.016	.013

TABLE 61 (Continued). FACILITY C CARBON ADSORPTION DATA

Parameter	Units	Day 1 Evaporator Condensate	Day 1 GAC Effluent	Day 2 Evaporator Condensate	Day 2 GAC Effluent	Day 3 Evaporator Condensate	Day 3 GAC Effluent
Other Analysis							
COD	mg/L	NS	NS	NS	17	NS	17
BOD_5	mg/L	NS	<6	NS	<6	NS	<20
TOC	mg/L	52	NS	52	<1	63	<1
Total Solids	mg/L	450	420	31	21	46	37
Total Dissolved Solids	mg/L	440	420	31	20	43	36
Total Suspended Solids	mg/L	10	2.8	<0.2	0.8	3.4	1.2
Total Volatile Solids	mg/L	410	340	31	18	39	18
Total Vol. Dis. Solids	mg/L	1.8	1.6	<0.2	0.8	2.4	1.2
Total Vol. Sus. Solids	mg/L	408	340	31	17	37	17

(710-850 ug/l), methyl ethyl ketone (230-360 ug/l for two days), diethylphthalate (547 ug/l for one day) and 2 nitrophenol (75-222 ug/l for two days) were reduced to below detection limits. The zinc measured in the influent samples for day 1 and day 3 was detected in the effluent at low concentrations. The solids in the wastes were primarily dissolved solids and the removal of these solids was low but for two of the three days the influent solids concentration was relatively low (30-50 ppm), thus, making high removal more difficult to achieve.